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DEGRADATION OF SLUDGES FROM HEAVY CRUDES IN CONTAINED REACTORS

Lloydminster Bioreactor for Sludge Degradation
[Part of the Program of the Federal Panel on Energy R&D (PERD)]

Initial Operation

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EXECUTIVE SUMMARY

A bioreactor for oil degradation was installed in a heated shed at Husky Oil's Lloydminster refinery the fall of 1989 and began running in January of 1990. The bioreactor contained API separator sludge and oil-contaminated diatomaceous earth which had been used as a filter press aid to break emulsions. After five months the numbers of oil degrading microbes remained high in the bioreactor although the frequency of organisms capable of degrading the bicyclic compound, naphthalene, was greatly decreased. This probably reflected the changing population dynamics in the bioreactor. At this time 45% - 60% of the oil was lost. Oil losses correlated with changed gas chromatographic patterns of the oil. Disposal options for the biodegraded reactor matrix include landfarming, landfilling and as an additive to improve the structure of sandy soils.

SOMMAIRE

Un réacteur pour la biodégradation du pétrole était installé à la raffinerie de Husky Oil à Lloydminster, AB. en automne de 1989. L'huile dans le réacteur était API separator sludge et diatomaceous earth contenant de l'huile. Après cinq mois les nombres des microbes qui peuvent dégrader l'huile restaient élevés dans le réacteur. Cependant, les microbes qui peuvent dégrader naphthalene etaient reduits. Peut-être c'est à cause d'un changement de la population microbienne. Après cinq mois 45% - 60% de l'huile était dégradé. La chromatpgraphie gaseuse a demonstré la dégradation de l'huile aussi. On peut amener les materiaux residuels dans le réacteur après la dégradation dans un landfarm ou un landfill ou le utiliser comme un additif pour ameliorer les sols sableus.

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1.0 OBJECTIVE

To monitor oil degradation in a contained bioreactor equipped with leachate recycle and located at Husky Oil's Lloydminster refinery.

2.0 INTRODUCTION

A field bioreactor containing tank bottom sludges adsorbed to Oclansorb heat-treated peat was installed at Husky Oil's Prince George refinery in the fall of 1988 (1). The reactor was in operation over the winter months and self-heated until the extreme temperature froze it. During this time, microbial numbers increased in the reactor and significant levels of oil were degraded (1). Bench scale reactors in the laboratory at NHRC containing sludge from conventional oil refining and heavy oil sludge sustained high numbers of oil degrading bacteria and over a 14 month period lost 62% - 78% of the oil (2). A demonstration bioreactor was erected at Lloydminster to assess degradation of two different oily sludges from a heavy oil refinery. This report details the work with the bioreactor at Lloydminster.

3.0 MATERIALS AND METHODS

3.1 Charging the Bioreactor

The bioreactor was constructed at Husky Oil's Lloydminster refinery. The bioreactor was constructed in two sections (Figure 1). One section contained Oclansorb and API separator sludge and the second section contained Oclansorb and filter press aid (oil-contaminated diatomaceous earth from a filter press trial used to break emulsions). Oclansorb, obtained from Hi Point Industries, Calgary, AB, was mixed with API separator sludge or the filter press aid in a mortar mixer until Oclansorb and the different oil sources became homogeneous mixtures. Potassium permanganate was added to each mixture as well as microbial culture taken from a landfarm and maintained at NHRC. The permanganate was added at a ratio of 1.4 g/kg of Oclansorb.

3.2 Microbial Analysis

Analyses of microbes in the reactors were performed by the most probable number technique (MPN). Samples from two depths at each sample plot were serially diluted in triplicate from 10^{-1}

to 10^{-12} in tubes of liquid culture medium and incubated for 14 days at ambient temperature. Turbid tubes indicating microbial growth were enumerated. These data were applied to standard probability tables published for this technique to estimate the number of bacteria per gram of solid material or per liter of leachate (3).

Minimal medium for enumeration of oil degrading organisms contained:

$K_2HPO_4.3H_2O$	13.1 g
$NaH_2PO_4.H_2O$	5.75 g
NH_4NO_3	2.0 g
$MgSO_4.7H_2O$	0.315 g
$CaCl_2.2H_2O$	0.0006 g
$FeSO_4.7H_20$	0.001 g
Distilled water	1 L

Final pH = 6.8

This medium was dispensed 4.5 mL per tube. After the tubes were inoculated, 50 microliters of Amauligak crude oil or 50 microliters of toluene containing 15 mg naphthalene were added to each tube of minimal medium. The toluene evaporated within 24 hours leaving crystals of naphthalene floating on the surface of the medium. Organisms able to metabolize the oil for growth caused an increase in turbidity and dispersed the oil. Organisms which grew on naphthalene also caused an increase in turbidity as well as the disappearance of the naphthalene crystals.

3.3 Water Analysis

The presence of polyaromatic hydrocarbons (PAH's) in the leachate of the bioreactor was performed by Core Laboratories, Calgary, Alberta. PAH's were analyzed by the EPA 625 method after extraction from the water with dichloromethane. Analysis was on an HP 5890 gas chromatograph and an HP 5970 mass spectrometer.

Elemental analysis of the leachate water and the "makeup" water was performed at Husky Oil's Lloydminster laboratory using procedures based on ASTM and standard laboratory methods. These procedures are included in Appendix 1.

Analysis for metals in the bioreactor matrices and sump was performed at Core Laboratories Ltd. in Calgary using Inductively Coupled Plasma - Atomic Emission Spectroscopy.

3.4 Extracted Oil

Samples (50 - 100 gram) of the Oclansorb/sludge or Oclansorb/oil-saturated diatomaceous earth matrices were removed from the reactor for oil extraction and analysis. Oil extraction was performed for six hours by the technique of Dean and Stark (Appendix 2) using hot toluene. Unlike other studies where hot dichloromethane was the extracting solvent (4, 25), toluene was used for extraction because it was assumed that with heavy oil, there would be few oil components more volatile than toluene likely to be stripped when the solvent was removed by evaporation.

The extracted oil was rotary evaporated to remove the toluene and the residue was weighed. The ratio of extracted oil to extracted oil plus solids was calculated. These ratios were compared to the ratio obtained on January 19, which is designated as zero time, enabling the calculation of percent oil loss over the period of operation. A 30 mg sample of the dried extracted oil was dissolved in 1 mL of carbon disulfide and subjected to gas chromatography in order to obtain a characteristic fingerprint of the oil and show how the patterns changed as degradation proceeded.

Gas chromatography was performed with a Perkin Elmer Sigma 2000 gas chromatograph equipped with an SPB-5 fused silica capillary column (30 m x 0.32 mm ID). The head pressure of the column was 75 kilopascals and the splitter gas flow was 30 mL/min. The purge gas flow was 3 mL/min. The injector and detector temperature was 320 degrees. The instrument was programmed to run at 70 degrees for ten minutes, then increase the temperature by 10 degrees per minute up to 100 degrees, followed by a 3 degree per minute increase up to 300 degrees. The temperature was maintained at 300 degrees for 30 minutes. The output of the resulting data was on an HP1000 Lab Data Acquisition System. A one microliter sample was used for injection.

3.5 Volatile Hydrocarbons

Volatile hydrocarbons were sampled using a Supelco Personal Air Sampler (PAS 3000). The sample intake was suspended 30 cm above the API separator sludge section of the bioreactor. The sampling rate was 0.5 liters/minute and sampling times were 30 and 60 minutes. Samples were drawn through small tubes containing Carbopack to adsorb the hydrocarbons. The hydrocarbons were subsequently recovered by thermal- desorption/cold-trap and analyzed on a gas chromatograph-mass spectrometer using a DB-1 capillary column suitable for C6 - C36 hydrocarbons.

The Carbopack was transferred into a glass tube which was heated to 300° C for ten minutes in the thermal desorption unit to volatilize the trapped hydrocarbons. A helium stream flushed the volatiles to a cold trap cooled to -170° C to condense them. The condensed volatiles were subsequently flash-heated to 270° C to introduce them into the capillary column in the gas chromatograph-mass spectrometer. The instrument was programmed to increase the temperature of the column from -20° to 300° C at 5° C/minute. The mass selective detector was scanned from 20 to 300 atomic mass units.

4.0 RESULTS

4.1 Bioreactor Set Up

The initial set up of the bioreactor was straightforward. Oclansorb was mixed with the API separator sludge, potassium permanganate and oil degrading microbial culture in a mortar mixer and placed in one side of the reactor. A similar mixture containing filter press aid (diatomaceous earth containing oil residue from a filter press trial to break emulsions) instead of sludge was placed in the other side of the reactor. Within a week of set up, the temperatures at Lloydminster dropped tremendously and the reactor froze. In January of 1990 the reactor was placed in a heated shed. The bioreactor thawed and was in operation for two weeks. The liner in the collection stand under the bioreactor developed a leak and water losses of 100 liters per day were seen. Additionally, the worker at the refinery assigned to check on the bioreactor and to take samples for analysis developed a rash on his hands. Refinery personnel attributed the rash to the microbes in the bioreactor and it was shut down until a Material Safety Data Sheet was prepared. (These interruptions caused six weeks of down time). When the bioreactor was restarted in March, the water was replenished daily (approximately 100 liters) until April 23. At this point the bioreactor was again shut in for three days to repair leaks. The reactor has run continuously since that time with no further difficulties.

4.2 Microbial Numbers

Although the bioreactor froze at one point, leaked and lost most of the original water, microbial numbers remained at a high level. Microbial counts taken at initial operation on January 19 indicated the number of oil degrading microbes was at 10^7 - 10^8 per gram of matrix both in the upper and lower layers of the bioreactor (Table 1). The numbers of organisms capable of degrading complex aromatic structures as represented by the bicyclic aromatic hydrocarbon, naphthalene, were at the level of 10^6 per gram of matrix (Table 2). Assessment of microbial numbers at three months and five months showed the total numbers of oil degraders to be stable (Table 1). However, by five months the number of organisms capable of degrading naphthalene had decreased to 10^3 - 10^4 per gram of matrix in all areas of the bioreactor (Table 2).

4.3 Temperature

The overall temperature in the bioreactor remained relatively constant. The five day average ambient temperature of the heated shed covering the bioreactor remained at 20° - 30° C during the run period of winter and early spring while the temperature of the reactor matrix remained around 20° C (Figure 3).

After the heater in the shed was turned off in the spring and early summer, the five day average temperature in the shed fluctuated with the external temperature and with the amount of solar radiation absorbed by the structure from 25° to 35°. In contrast, the five day average temperatures in the bioreactor bed showed minimal fluctuation between 20° - 25° during the same time interval (Figure 3). This may be attributed to the relatively cool and constant temperatures of the sump which was embedded in the earth beneath the bioreactor and probably acted as a heat exchanger for the unit.

4.4 Water Analysis

The pH of the leachate water in the sump was consistently lower than that of the feed water used to "top up" the sump due to evaporation and water loss by leakage (Table 3).

The water in the sump had higher concentrations of sodium, magnesium, iron, potassium, manganese, ammonia, chloride, sulfate, nitrate and phosphate than the feed water. The level of bicarbonate in the sump increased over the period of study while the level in the feed water was more constant (Table 4).

PAH's in the leachate water did not increase above the detection limit of 1 microgram/liter (Table 5).

Analysis for 28 metal elements (Table 6) showed slightly higher metal levels in the API separator sludge matrix than in the filter press aid matrix, although these differences may not be as significant. The sump water generally did not have individual metal levels above 1 ppm. All metals were substantially below the levels for hazardous wastes permitted by the Province of Alberta (Table 6).

4.5 Hydrocarbon Content

The toluene extractable hydrocarbon content of the bioreactor decreased over the period of operation. There were fluctuations in the oil levels over time but a general decrease was obtained at all locations in the bioreactor. Generally oil losses were in the range of 45% to 60% after four and a half months operation (Figure 4). The percentage of oil lost in the API separator sludge section of the bioreactor was 45% in both the upper and lower samples. The percentage of oil lost in the filter press aid section was 55% in the lower regions and 60% in the upper regions (Figure 4). Actual weights of oil recovered at various sampling points in the bioreactor are presented in Appendix 3.

Gas chromatograms of the January 19 recovered oil revealed the oil retained in the filter press aid was much lighter than that in the API separator sludge. The former eluted within 60 minutes, before the internal C-20 standard eicosane, while the latter eluted as a broad peak over 120 minutes (Figure 5). After treatment in the bioreactor, the oil in the filter press aid showed losses of specific peaks representing alkane molecules (Figure 6). The API separator sludge showed more dramatic changes. Specific peaks representing saturate alkane molecules disappeared. In addition, the bulk of the material which eluted as one unresolved hump over two hours in the chromatograms of the January samples, flattened out leaving two or three humps of unresolved material evident in the June samples. These humps eluted at 26 - 33, 46 - 52 and 78 - 86 minutes of the chromatogram (Figure 7). Gas chromatograms of the oil recovered from each sampling spot over the operation period are presented in Appendix 4.

4.6 Volatiles from the Bioreactor

A concern of a bioreactor is the release of volatile hydrocarbons. Examination of the volatile hydrocarbons trapped in 30 litres of air from above the bioreactor revealed the presence of 20 identified components in January shortly after reactor start up (Figure 8). In the Fall there were no detectable volatile hydrocarbons in the air above the bioreactor (Figure 9).

4.7 Disposal of Bioreactor Matrix

The residual matrix material from the bioreactor could be applied to a landfarm or it may be landfilled for ultimate disposal. Materials cannot be disposed of in a municipal landfill in Alberta if they contain any of the volatile compounds listed in Table 7 at concentrations greater than 1,000 mg/Kg. Toluene and xylenes were among the volatiles present in 30 litres of air from above the bioreactor in the January sampling but were absent in the Fall (Figures 8 and 9). The concentration of these restricted components was not determined.

5.0 DISCUSSION

5.1 Microbial Numbers

The number of oil degraders in the bioreactor appeared to be stable at 10^7 - 10^8 organisms per gram of matrix over the period of study. However by six months' operation the number of naphthalene degraders had severely decreased. This result is similar to that obtained in another study where a microbial population degrading heavy oil sludge contained few organisms capable of degrading naphthalene. However a large proportion of that population, when grown on Amauligak crude oil, produced water soluble colored compounds reminiscent of colored intermediates produced by microbial growth on polyaromatic compounds (4). This implies the population was acting on aromatics in the oil other than naphthalene. It is possible the naphthalene and similar components in the sludge had been degraded by the time of sampling and organisms capable of degrading them, being no longer viable were supplanted by other species.

There is accumulating evidence that organisms which degrade multiple ring compounds are somewhat selective in their substrate range. Generally, aromatic degraders do not attack the saturate fraction of the oil (5). Also, according to some reports, organisms which degrade four ring compounds do not attack three or two ring compounds as effectively (5,6). A culture isolated from beneath an oilfield degraded three and four ring compounds without a lag but required a two day lag to degrade the two ring compound naphthalene, suggesting that the ability to degrade the latter is a separate inducible system (16).

5.2 Reactor Temperature

Once the bioreactor was placed in a heated shed, there were no more problems of the matrix freezing. The temperature of the matrix paralleled the temperature in the shed. This contrasts with the bioreactor at Price George which ran several degrees above ambient during the first season (1). However the type of oil used in the bioreactor may dictate the rate of heat generation through oil degradation. The amount of heat generated will reflect the ease of oil degradation. An oil high in saturate materials will be more readily attacked than oil containing complex molecules. Higher metabolic rates will lead to more noticeable heat generation. Bench scale reactors in the lab at NHRC containing heavy oil sludge maintained their temperature at or near ambient while a bioreactor containing conventional crude oil sludge consistently had temperatures a few degrees above ambient (2,4). Lack of heat evolution in the bioreactor at Lloydminster may be due to the effective cooling by the sump water located in the earth under the reactor unit and which acted as a heat exchanger.

5.3 Water Analysis

The pH of the aqueous fraction in the bioreactor was always lower than that of the feed water. This may be due to leachates from Oclansorb which are slightly acidic. Oclansorb and raw peat applied to test plots at Husky Oil's Prince George landfarm lowered the pH of the soil from 6.86 to 5.84 - 5.57 (8). The acidity may be attributed to humic and fulvic acids present in peat (18,19) which give it a pH of 5 - 6 (9,17). However, the leachate from the bioreactor was not assayed for these acids. Increased acidity of the aqueous fraction may also be attributed to acid metabolites from biodegradation of oil components. The linear alkanes in the oil are oxidized to the corresponding alcohols and subsequently to the fatty acids. It is in this form that they are assimilated by the degrading organisms. Aromatic and other ring structures are also converted to acid intermediates after ring cleavage (9, 10, 11, 15). These acid intermediates could contribute to the acidification of the sump water.

The source of initially increased concentrations of sodium, potassium and chloride ions in the sump compared to the feed water is attributed to the oily sludge itself. Concentrations of sodium and chlorides at 2000 ppm are not uncommon in desalter wash water at refineries (B. Faber, Husky Oil Ltd., personal communication). Subsequent decrease in the chloride ion content may be attributed to sequestering by the matrix in the bioreactor or to dilution and leaks. Initially increased concentrations of potassium and phosphate ions can be attributed to the fertilizer added to the bioreactor. This slow release fertilizer is a commercial blend sold by Beaver Lumber containing 7% phosphorus, 7% nitrogen and 7% potash. The relatively constant level of sulfate in the sump may be indicative of slow release sulfates present in the original sludge. Such sulfates might be in the form of precipitates of calcium as the solid bioreactor matrix was found to contain high calcium levels (Table 6).

Permanganate had been added to the bioreactor but no pink color of permanganate was observed in the sump water indicating reduction of this species. The levels of manganese present in the sump were relatively low although higher than in the feed water. Perhaps the manganese ions were retained by the reactor matrix since peats are known to retain metal ions (18,19).

The concentration of bicarbonate in the feed water remained relatively constant throughout the study period while it gradually increased in the sump. Increased bicarbonate in the sump may have originated from carbon dioxide produced by microbial metabolism.

The overall concentration of PAH's in the leachate was low. It may be difficult to quantify this material in the leachate fraction because of changes in volume of the water fraction due to evaporation and "topping up" over the course of the study. Concentrations of PAH's are generally in the order of tens to hundreds of milligrams per kilogram of oily sludge (13). Lloydminster heavy oil is estimated to contain 250 - 400 mg/L PAH's (14). Solubilities of PAH's in water are in the order of 0.004 to 30 mg/L. Since PAH's are not believed to be readily degraded and therefore should accumulate, their concentration in the water fraction should increase as other components in the oil are degraded. Lack of PAH accumulation in the reactor leachates indicates they are being biodegraded. This agrees with observations previously made with the laboratory bioreactors (2,4).

Metal analysis indicated there are a variety of metals in the bioreactor matrices. The origin of these metals would be the produced brine, heavy oil, and refinery process. Analysis of produced waters by Inductively Coupled Plasma - Atomic Emission Spectroscopy from the Furness field in Lloydminster gave relatively high concentrations for calcium, magnesium, potassium, and sodium (23) as was seen in the bioreactor matrices. Heavy oil is known to contain heavy metals such as nickel and vanadium in concentrations compatible with the results in Table 6 (24). The other metals will come from the refinery process. Peat is known to have ion exchange capacity (18,19) and will most likely have concentrated the metals during the percolation process. The results of the sump sample show these metals are not leaching off the matrices into the water as biodegradation occurs.

Comparison of the metal contents of samples from the Oclansorb/sludge matrix, the Oclansorb/filter aid matrix and the sump with the Province of Alberta standards for heavy metals (26) (Table 6) indicate the metal contents in both bioreactor matrices would pass the criteria for landfill disposal (Appendix 5). This material could therefore be landfilled or landfarmed. Options for water disposal at refineries include municipal disposal, on-site disposal ponds, water recycle and deep well injection. Husky's Lloydminster operations use the latter option extensively. The low levels of metals and PAH's present in the leachate water from the bioreactor suggests its compatibility with any of these disposal methods. It is recognized the slop oil is continually changing so metal levels in the matrix and in the leachate would have to be established for each run of the bioreactor.

5.4 Hydrocarbon Content

Significant oil losses occurred in the bioreactor over the period of operation. A larger percentage of oil was apparently lost from the section of the bioreactor containing the filter press aid than from the section containing API separator sludge. However, since the filter press aid section of the bioreactor initially contained less oil than the API separator sludge section, comparable oil losses in that section will represent a larger percentage of its oil content. Oil losses of 45% - 60% after five months operation compare with losses of 70% after eight months' operation of a bioreactor degrading sludge from conventional oil refining at Prince George (25). The Prince George bioreactor was frozen during part of the eight month operation period indicating the rates of oil degradation in both reactors may be quite similar.

The quality of the oil in the filter press aid is distinctly different from that in the API sludge. The former was fully eluted within 50 minutes during gas chromatography while material from the API sludge was eluted over 120 minutes. A C-20 eicosane standard was placed in both oil samples and was found consistently with the same retention time in all samples. The oil containing smaller molecular weight species will be more readily degraded and will contribute to a faster degradation rate in the section of the bioreactor containing the filter press aid.

The gas chromatograms of the oil recovered from both sections of the bioreactor showed significant losses of the lighter oil components which are the more easily degraded fractions of the oil (12,22). Partial resolution of the oil from the API separator sludge side of the reactor into three humps after microbial attack further attests to biodegradation of the oil with the residual material representing the more recalcitrant oil fractions.

5.5 Volatiles from the Bioreactor

The bioreactor releases volatile hydrocarbon components at least in the start up phase. These may constitute a health concern. However, since the composition is the hydrocarbons in the bioreactor is the same as in the API separator tank, health concerns should not be an increased issue at the refinery site. As a precaution, further tests should be performed to ascertain the levels of the individual volatile hydrocarbons released from the bioreactor.

5.6 Disposal of Bioreactor Matrix

Disposal of the bioreactor matrix can be by landfarming or by landfilling. The method of disposal will depend on the quantity of land available for landfarming and the rules for landfilling in a particular province or locality. In Alberta landfilling of hydrocarbon materials is allowed at approved sites with defined limits for a variety of contaminants (Appendix 5). Based on preliminary results it appears that the restricted volatiles are not present in the biodegraded matrix but more rigorous tests will have to be performed to quantify the results. In Saskatchewan, hydrocarbon materials may not be landfilled. However, exceptions to these regulations may be obtained by special order of the Minister of Environment after certain conditions/standards have been met. These are determined for each individual case after thorough examination of the proposed landfill materials.

Disposal of the bioreactor matrix in a municipal landfill has been the proposed method of disposal because of the convenience, i.e., the low economic cost, established sites and transport, proximity to sludge generation sites and optimal bioreactor location.

A novel disposal technique for the bioreactor matrix may be as an additive to improve soil structure. Agriculture Canada is conducting ongoing research to utilize oily waste sludge to conserve and improve the structure of sandy cultivated soils and improve their humic content (20). Results obtained over a three year test period show sludge incorporation greatly improved the soil's structure through large and persistent increases in primary and secondary aggregation. It also effected beneficial increases in soil microbial activities and populations. Humic materials were also increased in the sandy soils (V.O. Biederbeck, personal communication).

A negative effect of employing hydrocarbon for this treatment was phytotoxicity to cereal grain production but this could be prevented by increased fertilization (20). Separate work conducted at the University of Calgary's Kananaskis Center for Environmental Research established that barley crops grown in soil containing oily sludge did not accumulate compounds attributable to the organics originating in the sludge (21). It thus appears that the technique of using oily sludge materials to aggregate soils does not pose an environmental threat. Interest has been expressed by Environment Canada in utilizing the bioreactor matrix to improve soil structure because of degraded oil content, the presence of the peat material as a soil additive and the increased microbial numbers present on the material which could improve soil fertility. It may effect soil structure improvements in a reduced time span without phytotoxic effects. This will be explored further with Environment Canada.

6.0 CONCLUSIONS

1. Over a five months period high numbers of oil degraders were maintained in a bioreactor for heavy oil degradation at Husky Oil's Lloydminster refinery.

2. The bioreactor maintained an average temperature of 20 - 25 degrees throughout the operational period.

3. PAH's or heavy metals did not accumulate in the leachate water.

4. Hydrocarbon content of the bioreactor decreased 45% - 60% over five months of operation.

5. Techniques for disposal of the biodegraded oily sludge matrix include landfarming, landfilling and as an agent for improving aggregation and fertility in sandy loam cultivated soil.

7.0 RECOMMENDATIONS

1. Running of the bioreactor should continue. It should be recharged with oily waste and monitoring continued.

2. Ultimate disposal of the spent matrix from the bioreactor should be investigated. Discussions should be continued with the Environment departments of Alberta and Saskatchewan and with Agriculture Canada.

3. Plans for a larger bioreactor to be built at Lloydminster should be formulated after consultation with the personnel at Husky's Lloydminster refinery.

4. Engineering aspects of the bioreactor in terms of oxygen transfer rates and heat fluxes should be investigated for scale up of the demonstration unit to commercial size.

5. Alternative matrices to Oclansorb should be assessed for their ability to absorb oily wastes and their effects on degradation of waste sludges.

6. Biodegradation of other waste streams in a bioreactor such as those generated by Husky Oil's upgrader as well as chlorinated hydrocarbon species should be assessed.

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9.0 TABLES

TABLE 1: OIL-DEGRADING MICROBES IN THE BIOREACTOR

(#/g, MPN METHOD USING AMAULIGAK OIL AS A SUBSTRATE)

LOCATION*		JANUARY 19/90	APRIL 16/90	JUNE 13/90
1	UPPER	2.8×10^7	3.0×10^8	5.0×10^8
	LOWER	2.8×10^6	5.0×10^8	5.0×10^8
2	UPPER	2.2×10^7	5.0×10^7	9.0×10^7
	LOWER	2.8×10^8	1.9×10^8	1.9×10^8
3	UPPER	9.0×10^7	5.0×10^8	1.9×10^7
	LOWER	2.2×10^8	9.0×10^8	5.0×10^9
4	UPPER	3.0×10^7	1.5×10^9	5.0×10^7
	LOWER	2.2×10^8	9.0×10^8	1.9×10^9
5	UPPER	2.2×10^8	5.0×10^8	9.0×10^7
	LOWER	2.2×10^8	9.0×10^8	1.9×10^7
6	UPPER	2.8×10^8	1.5×10^9	5.0×10^7
	LOWER	2.8×10^8	5.0×10^9	5.0×10^7

*Samples were taken at depths of 30 cm (upper) and 60 cm (lower) in the bioreactor matrix (Figure 2). Four sites (2-5) were in the API Separator Sludge matrix and two sites (1,6) were in the Filter Press Aid matrix.

TABLE 2: COMPLEX AROMATIC DEGRADING MICROBES IN THE BIOREACTOR

(#/g, MPN METHOD USING NAPHTHALENE AS A SUBSTRATE)

LOCATION*		JANUARY 19/90	JUNE 13/90
FILTER PRESS AID	UPPER	3.0×10^5	8.0×10^3
	LOWER	1.9×10^6	5.0×10^3
API SLUDGE	UPPER	9.0×10^6	1.9×10^4
	LOWER	1.5×10^6	3.0×10^4

*Samples (1 gram/site) were taken to make composite samples of both matrices at the upper and lower levels. A half gram sample of the composite was used for the MPN sample.

TABLE 3: pH OF THE LEACHATE WATER

DATE	pH	
	Feed (make up)	Sump
Jan 19	7.68	6.68
March 19	7.52	6.49
April 2	7.51	6.97
April 16	7.59	7.49
May 28	8.30	7.18
June 13	8.16	7.49

TABLE 4: CATION AND ANION ANALYSIS OF THE BIOREACTOR WATER

A. WATER ANALYSIS FOR THE BIOREACTOR SUMP

DATE	ION CONCENTRATION (mg/L)												
	Na^{+}	K^{+}	NH_4^{+}	Mn^{+2}	Ca^{+2}	Mg^{+2}	Fe^{+3}	Cl^{-1}	HCO_3^{-1}	CO_3^{-2}	NO_3^{-1}	SO_4^{-2}	PO_4^{-3}
January 19	405	455	347	2	69	52	0.2	953	366	0	ND	1103	151
March 19	440	121	ND	4	102	77	1.3	136	380	0	203	844	59
April 2	370	77	ND	3.4	85	61	2.8	107	385	0	65	787	77
April 16	415	73	ND	2.8	78	60	2.5	114	424	0	ND	854	68
May 28	330	49	ND	2.6	79	59	2.2	77	541	0	8	603	29
June 13	470	68	ND	3.8	103	85	0.9	103	634	0	4	958	35

B. WATER ANALYSIS FOR THE BIOREACTOR FEED WATER

DATE	ION CONCENTRATION)												
	Na^+	K^+	NH_4^+	Mn^{+2}	Ca^{+2}	Mg^{+2}	Fe^{+3}	Cl^{-1}	HCO_3^{-1}	CO_3^{-2}	NO_3^{-1}	SO_4^{-2}	PO_4^{-3}
January 19	282	7	3.2	0.4	104	48	0.5	51	551	0	ND	530	ND
March 19	296	8	ND	0.3	74	45	0.5	45	536	0	0	490	0
April 2	288	7	ND	0.4	82	43	0.3	63	517	0	ND	535	ND
April 16	296	7	ND	0.5	81	45	1.3	65	546	0	ND	522	ND
May 28	10	39	ND	0.03	31	12	0.3	8	98	0	ND	57	ND
June 13	320	8	ND	0.1	45	42	0.5	67	444	0	ND	564	ND

ND: Not Determined

TABLE 5: PAH's IN THE LEACHATE WATER

COMPONENT	CONCENTRATION (ug/L)*
Naphthalene	<1
Acenaphthylene	<1
Acenaphthene	<1
Fluorene	<1
Phenanthrene	<1
Anthracene	<1
Fluoranthene	<1
Pyrene	<1
Benzo(a)anthracene	<1
Chrysene	<1
Benzo(b)fluoranthene	<1
Benzo(k)fluoranthene	<1
Benzo(a)pyrene	<1
Indeno(1,2,3-cd)pyrene	<1
Dibenzo(a,h)anthracene	<1
Benzo(g,h,i)perylene	<1

*A detection limit of 1 microgram/L was used based on a water sample of 1 L.

TABLE 6: TRACE METAL ANALYSIS OF BIOREACTOR (mg/kg or mg/L)*

ELEMENT	SAMPLES			PROVINCIAL** LIMITS
	SLUDGE	FILTER AID	SUMP	
ALUMINUM	530	490	0.17	-
ARSENIC	<10	<10	<.1	500
BARIUM	110	99	0.079	-
BERYLLIUM	<1	<1	<.001	-
BORON	<10	<10	0.83	-
CADMIUM	0.6	<0.5	<.002	100
CALCIUM	9900	5600	250	-
CHROMIUM	10	7	0.006	500
COBALT	<3	<3	<.005	-
COPPER	14	16	0.004	-
IRON	3500	3000	1.8	-
LEAD	15	7	<.03	500
MAGNESIUM	1200	1100	150	-
MANGANESE	330	220	5	-
MERCURY	0.015	0.017	<.001	100
MOLYBDENUM	<5	<5	0.011	-
NICKEL	12	6	0.02	134
PHOSPHORUS	1200	840	9.7	-
POTASSIUM	300	200	91	-
SELENIUM	<5	<5	<.05	100
SILVER	<1	<1	<.005	-
SODIUM	720	640	940	-
STRONTIUM	110	95	3.4	-
THALLIUM	<10	<10	<.1	130
TIN	<5	<5	<.02	-
TITANIUM	30	26	<.002	-
VANADIUM	20	<10	0.004	300
ZINC	48	48	0.079	-

*August 15, 1990 sample.

**Beyond these concentrations, the material is considered as hazardous waste in Alberta (26) (Appendix 5).

TABLE 7: COMPOUNDS RESTRICTED FROM LANDFILLS*

- Acetone
- Benzene
- n-Butyl alcohol
- Carbon disulfide
- Carbon tetrachloride
- Chlorobenzene
- Cresols and cresylic acid
- Cyclohexanone
- Ethyl acetate
- Ethyl benzene
- Ethyl ether
- Isobutanol
- Methanol
- Methylene chloride
- Methyl ehtyl ketone
- Methyl isobutyl ketone
- Nitrobenzene
- 2-Nitropropane
- Pyridine
- Tetrachloroethylene
- Toluene
- 1,1,1, and 1,1,2-trichloroethane
- Trichloroethylene
- Xylene

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*From Reference 26, Appendix 5.

10.0 FIGURES

FIGURE 1: The Bioreactor

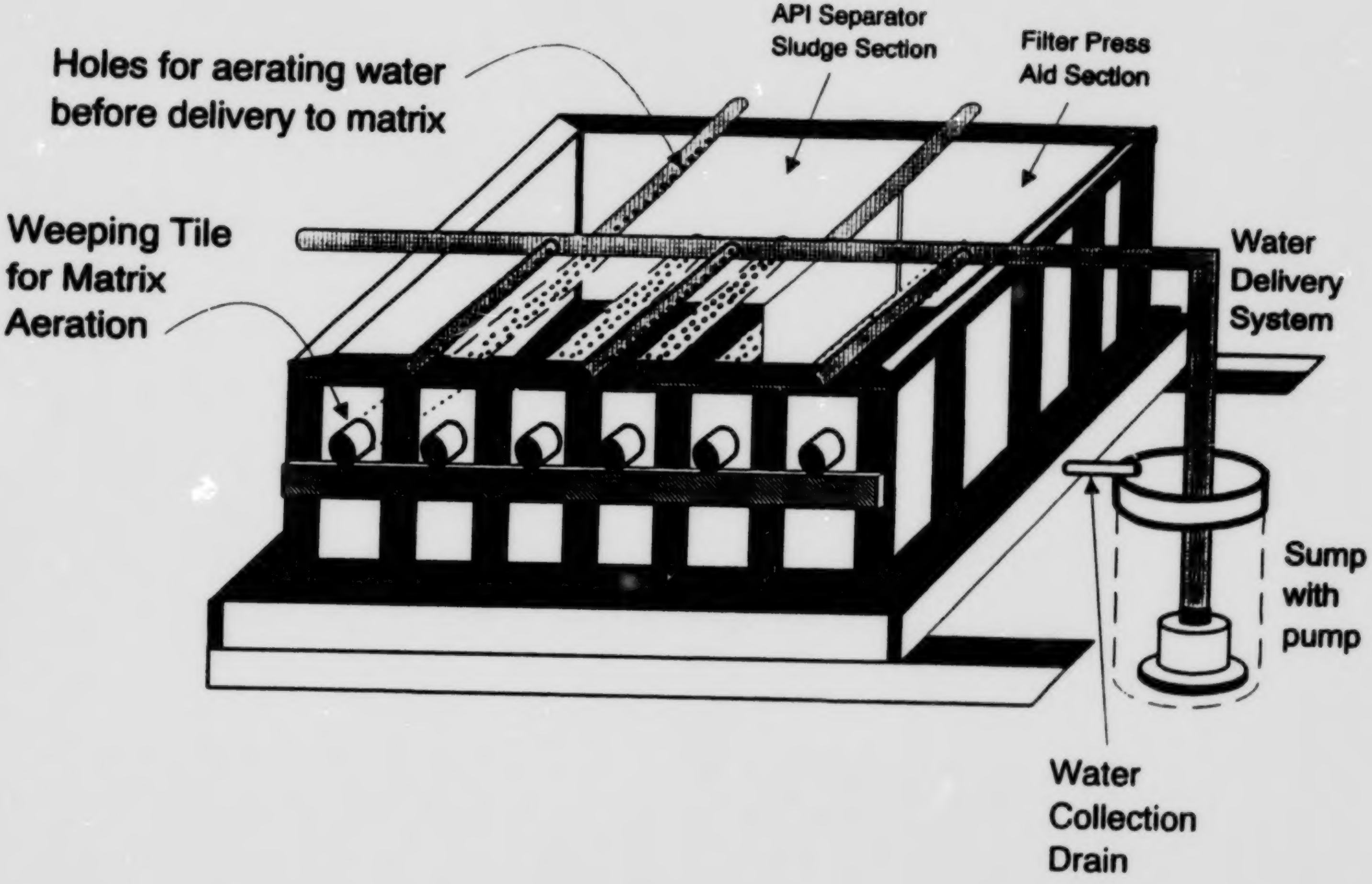

Holes for aerating water before delivery to matrix
API Separator Sludge Section
Filter Press Aid Section
Weeping Tile for Matrix Aeration
Water Delivery System
Sump with pump
Water Collection Drain

FIGURE 2: Sampling Points in the Bioreactor

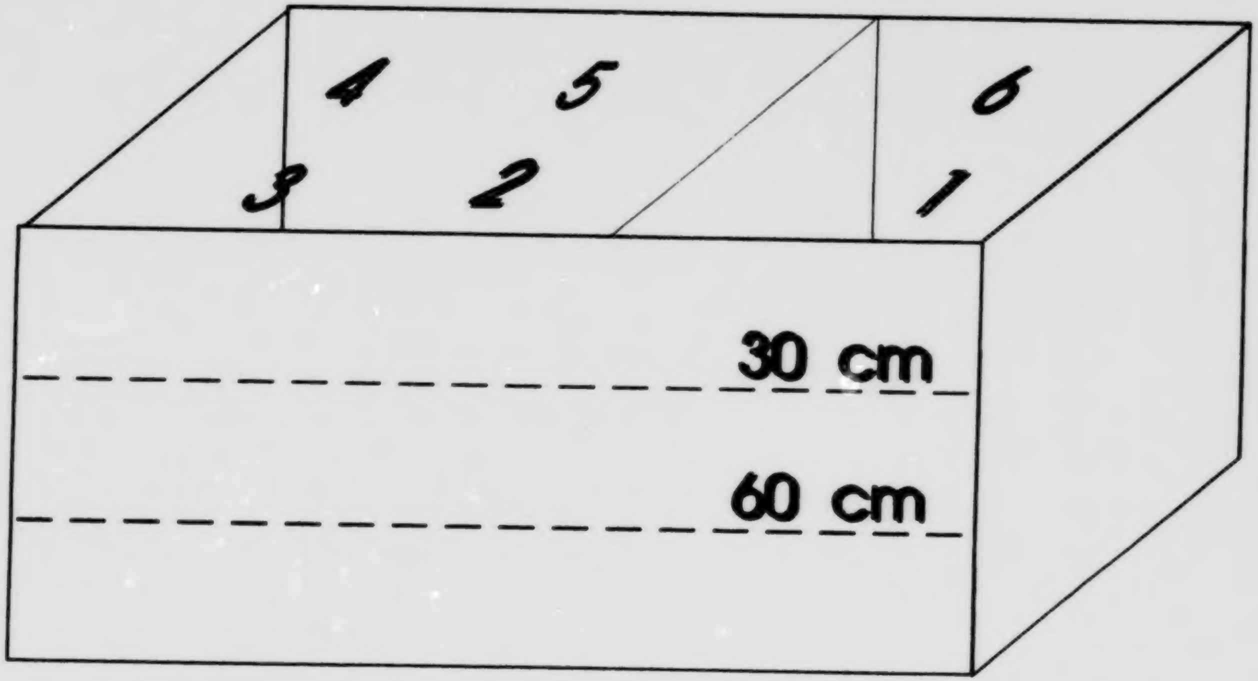
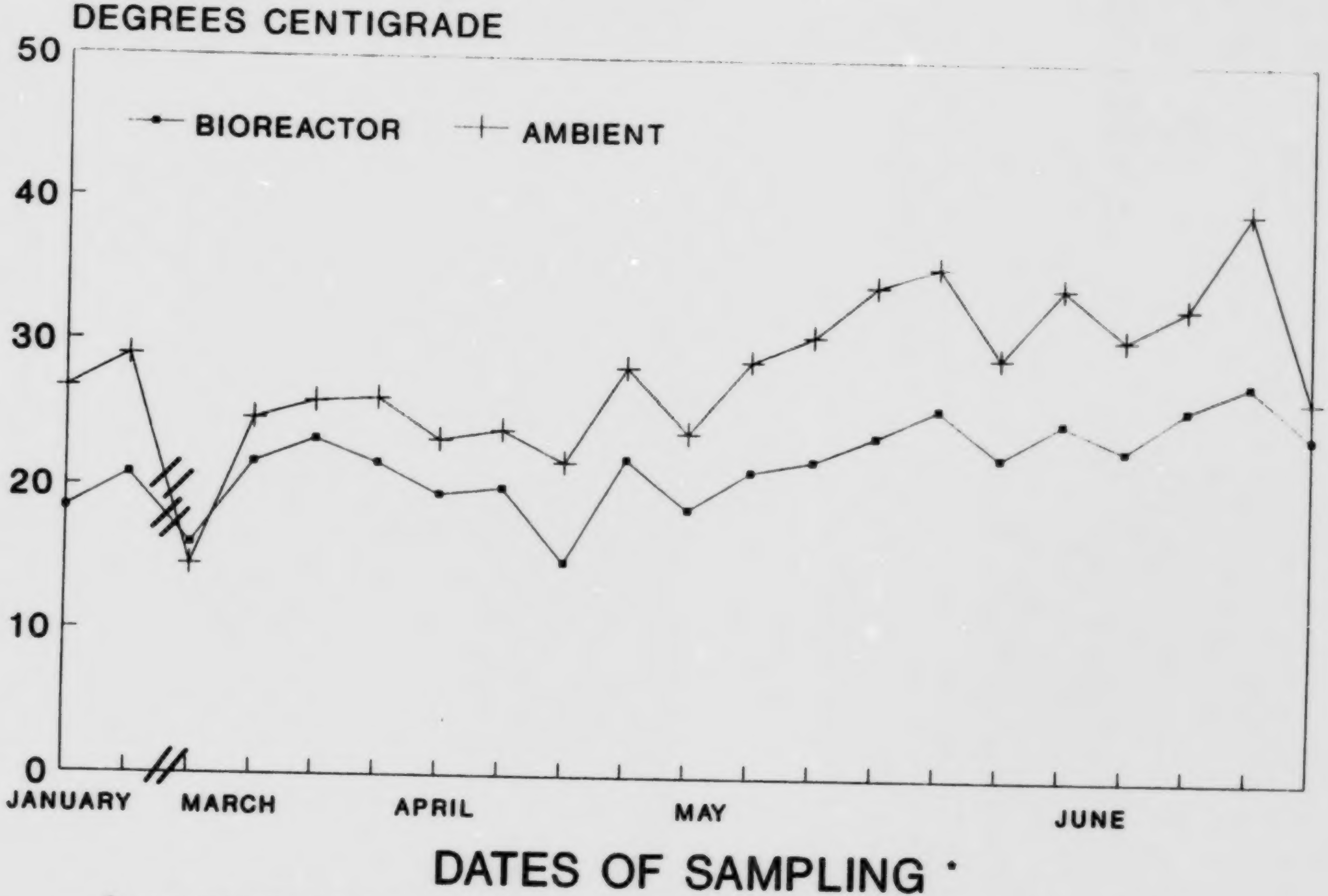


FIGURE 3 FIVE DAY AVERAGE TEMPERATURES IN THE BIOREACTOR AND IN THE COVERING SHED



* NO SAMPLES FROM THE END OF JANUARY TO THE MIDDLE OF MARCH

FIGURE 4 PERCENT OIL LOSS FROM THE BIOREACTOR

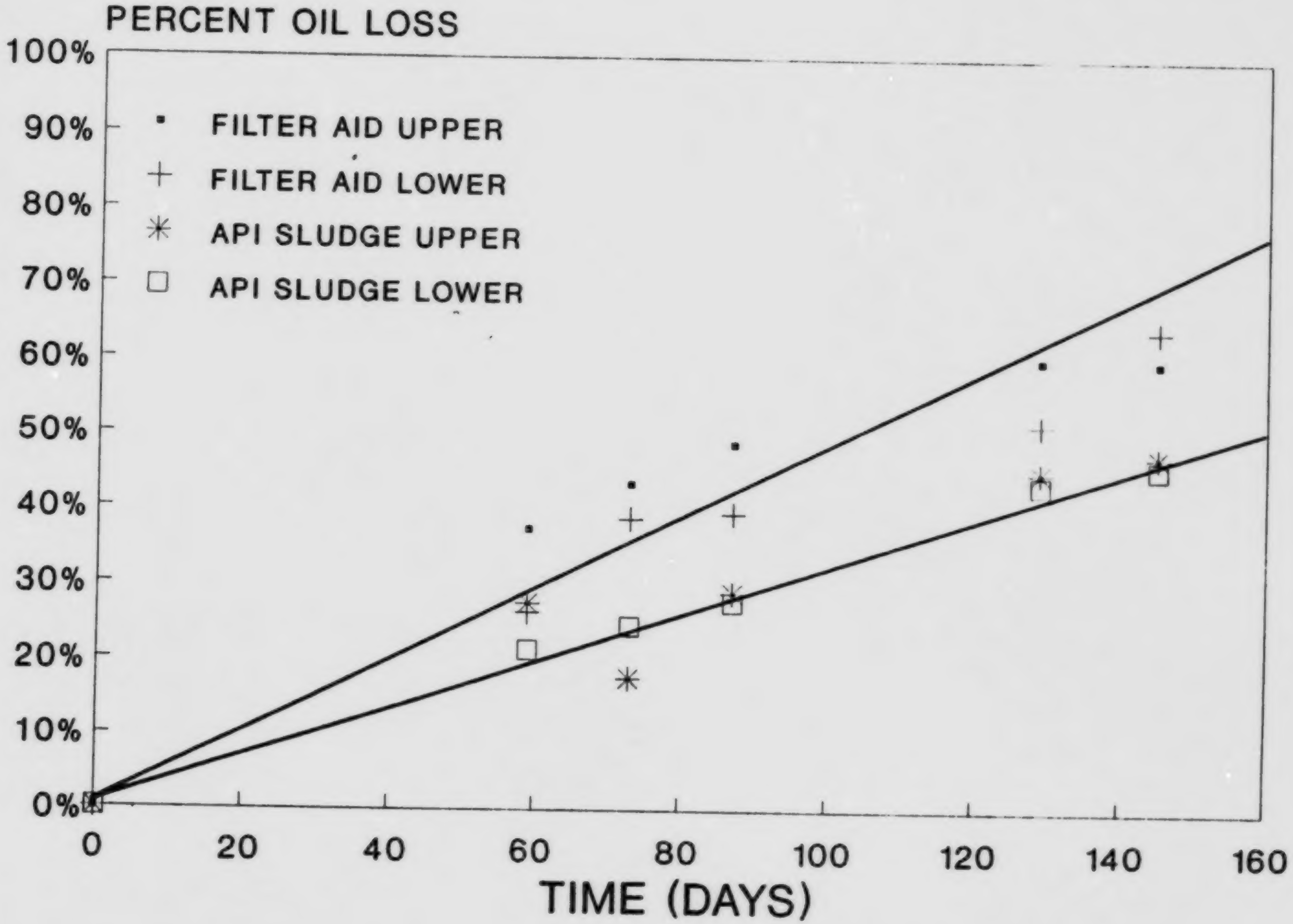


FIGURE 5

COMPARISON OF THE GC OF THE OIL IN API SEPARATOR SLUDGE AND IN THE FILTER PRESS AID

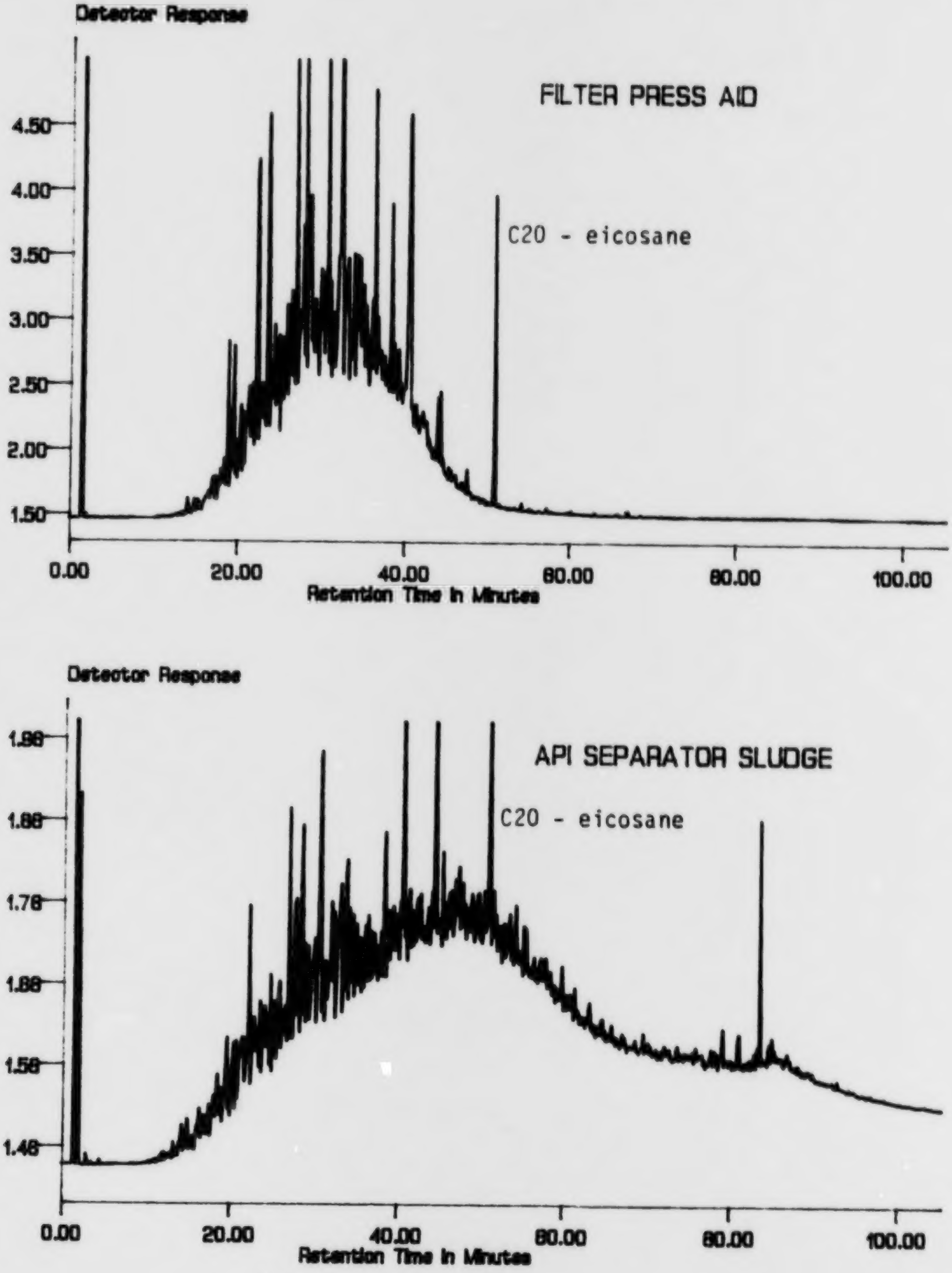


FIGURE 6. CHANGING GC PATTERNS OF THE OIL RECOVERED FROM THE SECTION OF THE BIOREACTOR CONTAINING THE FILTER PRESS AID

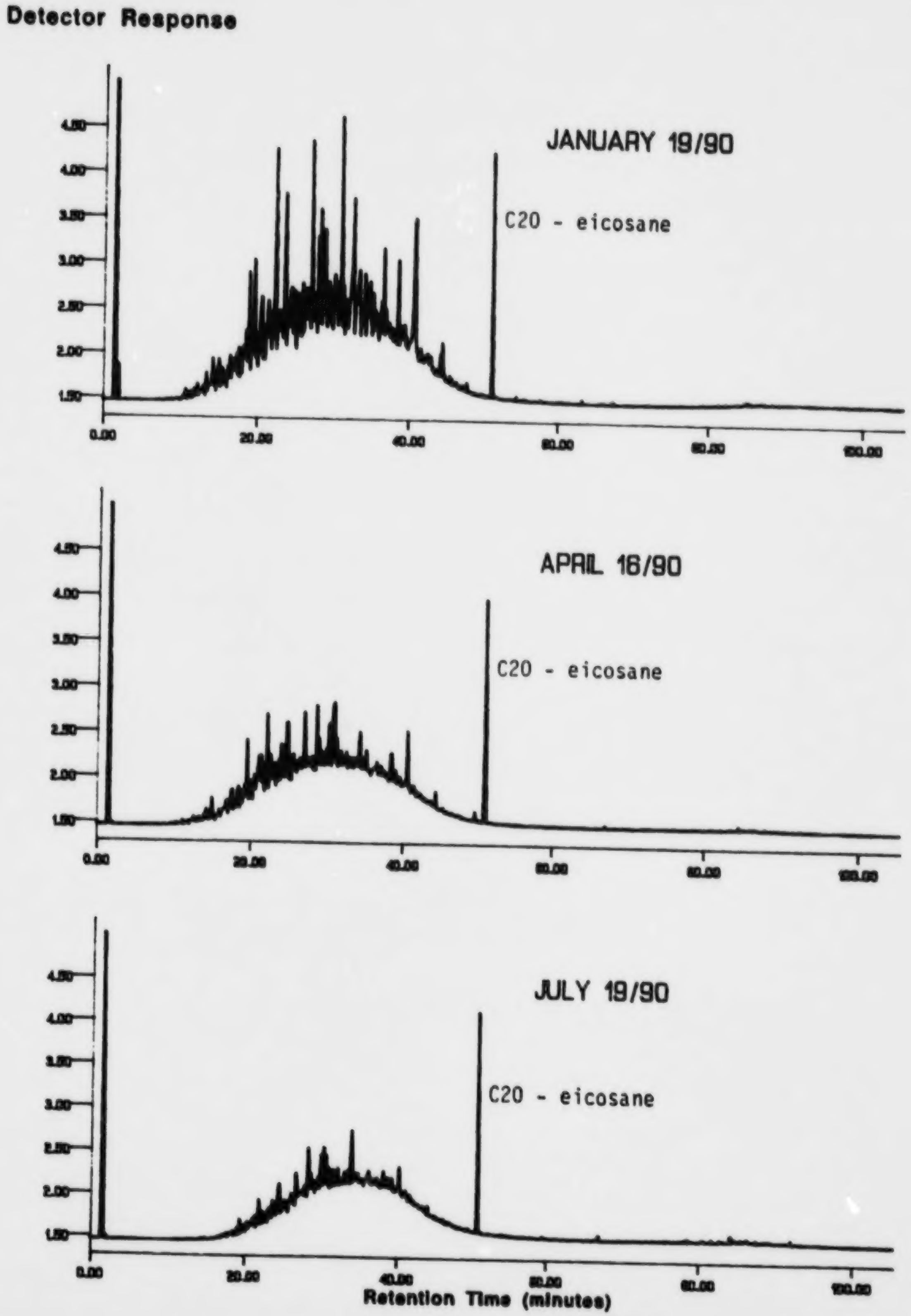


FIGURE 7. CHANGING GC PATTERNS OF THE OIL RECOVERED FROM THE SECTION OF THE BIOREACTOR CONTAINING API SEPARATOR SLUDGE.

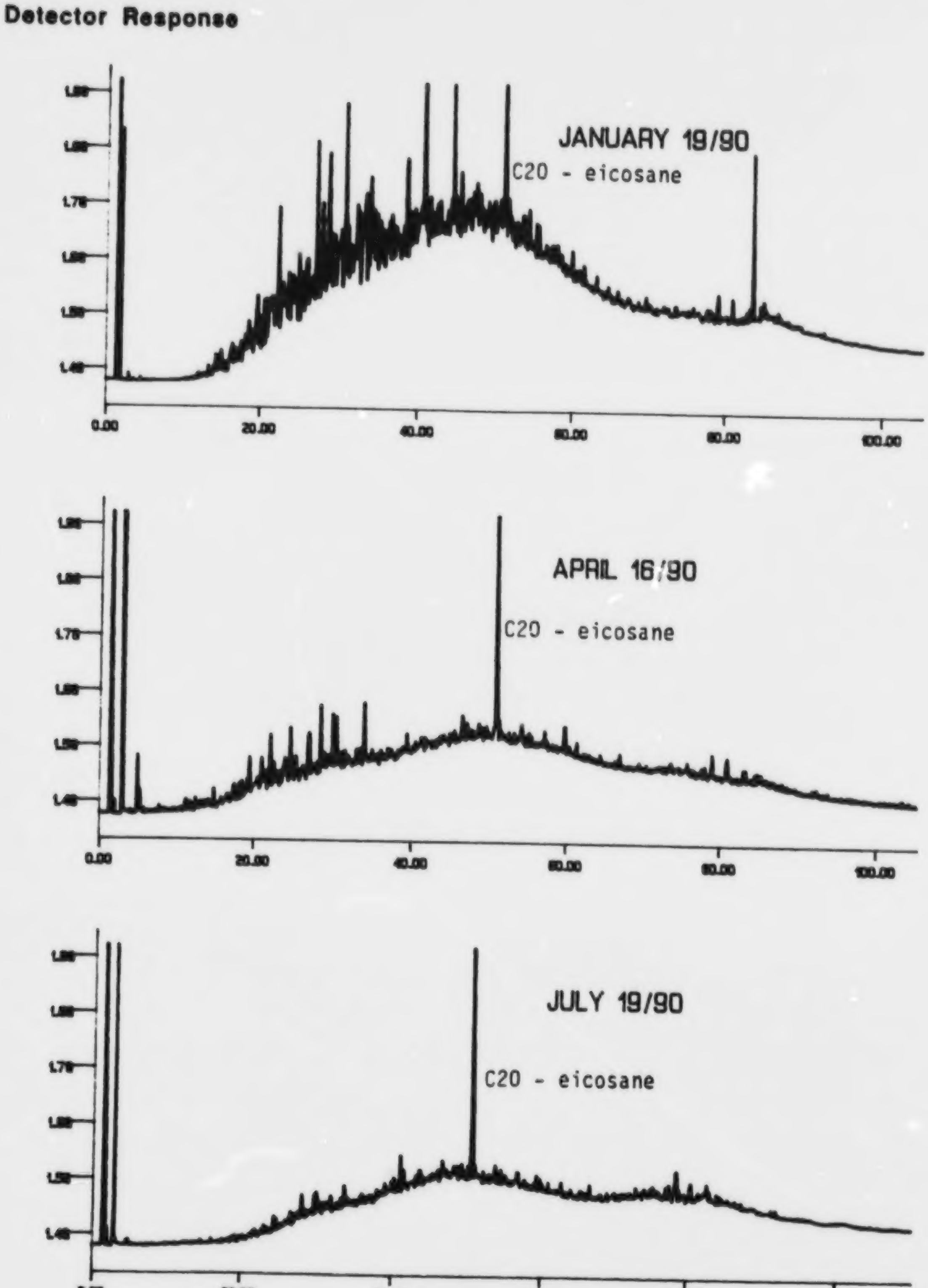


FIGURE 8: GC PATTERN OF THE VOLATILES FROM THE BIOREACTOR ON JANUARY 19, 1990

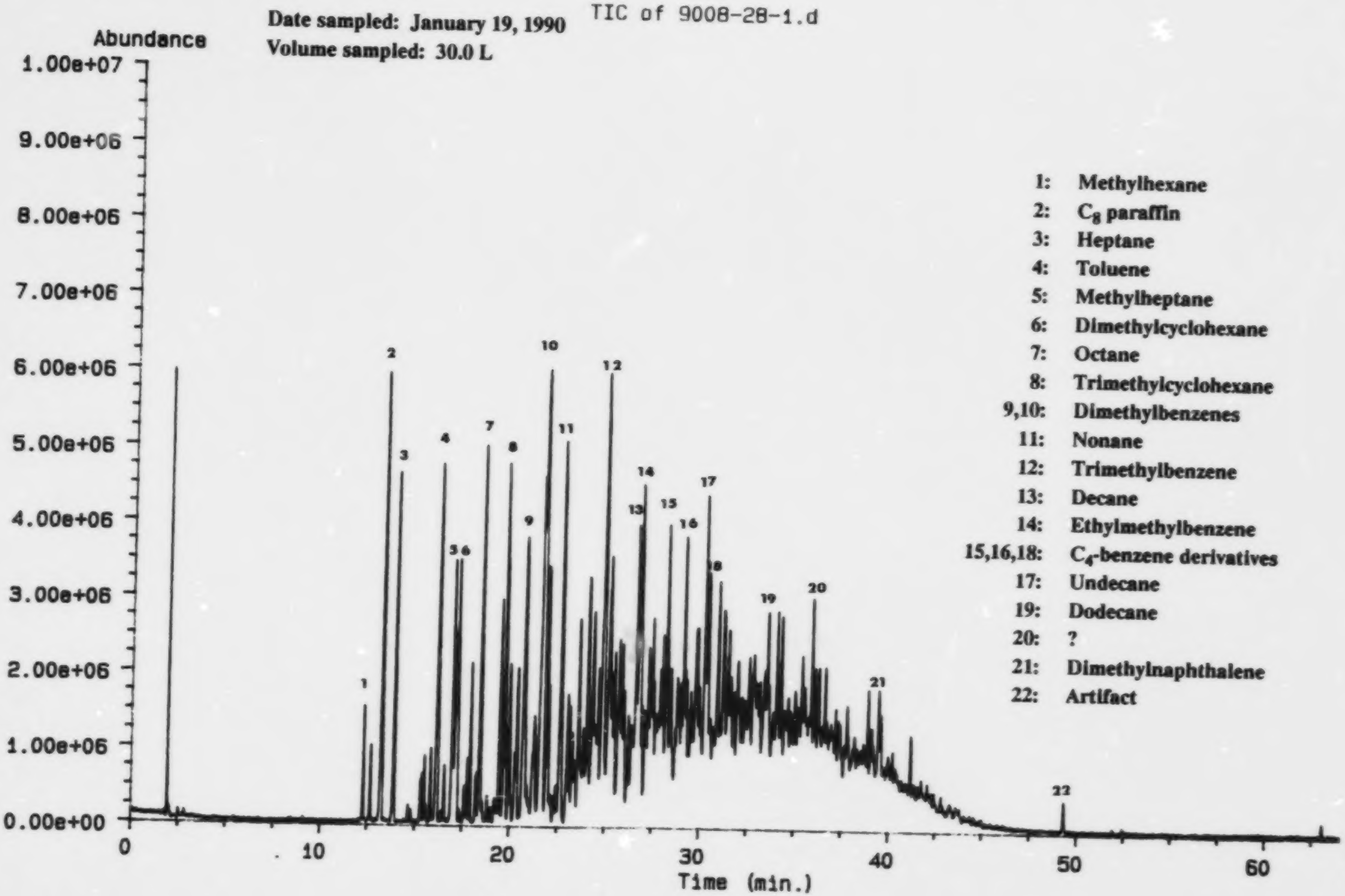
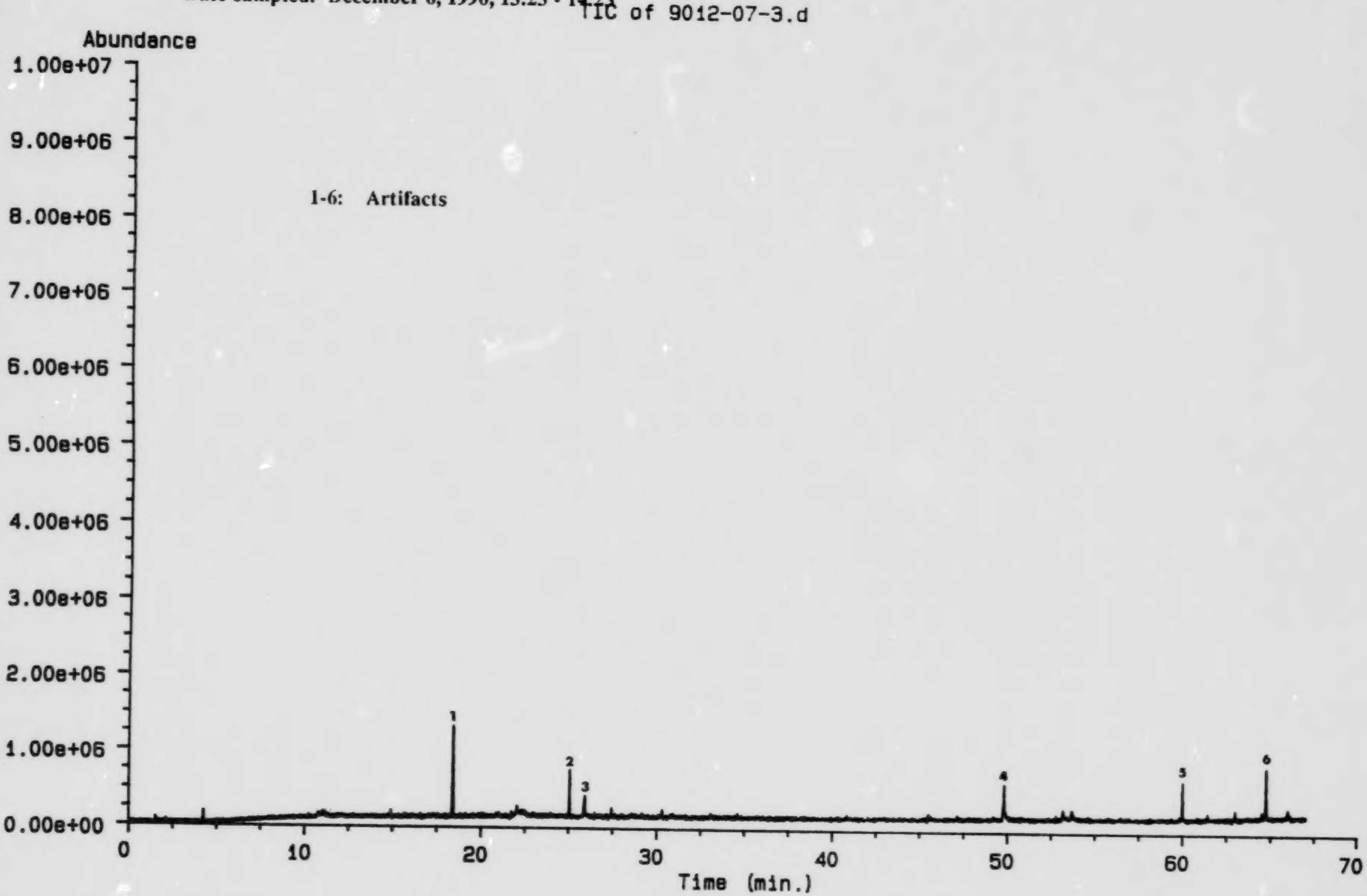


FIGURE 9: GC PATTERN OF THE VOLATILES FROM THE BIOREACTOR IN DECEMBER, 1990

Date sampled: December 6, 1990, 13:23 - 14:23



APPENDIX 1. METHODS OF WATER ANALYSIS

METHOD P9

STIFF PATTERN

A) **METHODS USED:**

✓	W13	PH
	02	Density of Crude Oil
✓	W18	Resistivity
✓	W17	Conductivity
—	W6, 6a, or 6b	Oils & Greases
—	W2	Total Suspended Solids
—	W8	Total Dissolved Solids
✓	W16	Cations By Emission
✓	W15	Cations By AA
—	W11	Cations By Ion Chromatograph
✓	W10	Anions By Ion Chromatograph
✓	W14	Alkalinity

N) **NOTES:**

1) See attached form.
2) Total Hardness and total dissolved solids are calculated normally.
3) These tests (W2, W6 and W8) are only done when we are specifically asked to do them.
4) The choice of oil & grease method used depends on the nature of the sample and why the analysis is being done. Field samples are usually done using method W6 (chloroform). Oils & greases done for environmental purposes are done using method 6a or 6b (freon). Very clean samples can be done using 6b (Horiba method).
5) We are capable of doing these elements on the AA or on the IC. The choice of method depends on availability of equipment.
6) The total MEQ/L balance is as follows:

$$\frac{\text{Cation MEQ} - \text{Anion MEQ}}{\text{Cation MEQ}} \times 100 \le \pm 10\%$$

Husky

HEAVY OIL LABORATORY
WATER ANALYSIS WORK SHEET I

WELL NAME	LOCATION	FILE REFERENCE NUMBER
ANALYST	DATE ANALYSED YEAR / MONTH / DAY	LAB SAMPLE NUMBER
FORMATION	FIELD	DATE YEAR / MONTH / DAY
DATE SAMPLED YEAR / MONTH / DAY	SAMPLED BY	SAMPLE CONTAINER(S)
		PRESERVATIVE(S)

OBSERVED pH @ ____ °C W13
DENSITY @ 15°C, g/ml 02
RESISTIVITY @ 25°C, ohm - cm W18
CONDUCTIVITY @ 25°C, µs/cm W17
OIL AND GREASE, mg/L W6 or W6a & W6b (Note #3 & 4)

TOTAL HARDNESS, mg/L Calculation (Note #2
TOTAL DISSOLVED SOLIDS, mg/L Calculation or W8
TOTAL SUSPENDED SOLIDS, mg/L W2 (Note #3) (Note
VOLATILE SUSPENDED SOLIDS, mg/L ____
FIXED SUSPENDED SOLIDS, mg/L ____

ION	CONCENTRATION		
	mg/L	FACTOR	meq/L
Na	Note 5 W16 or W11	× 0.0435 =	
Ca	Note 3 W15 or W11	× 0.0499 =	
Mg	Note 5 W15 or W11	× 0.0823 =	
Fe	W15	× 0.0537 =	
K	Note 5 W16 or W11	× 0.0256 =	
Ba		× 0.0146 =	
Sr		× 0.0228 =	
Mn	W15	× 0.0364 =	
Si		× 0.1424 =	
TOTALS			NOTE 6

ION	CONCENTRATION		
	mg/L	FACTOR	meq/L
Cl	W10	× 0.0282 =	
HCO_3	W14	× 0.0164 =	
SO_4	W10	× 0.0208 =	
CO_3	W14	× 0.0333 =	
OH	W14	× 0.0588 =	
TOTALS			NOTE 6

TOTAL HARDNESS CALCULATIONS

TOTAL HARDNESS = [mg/L calcium] ____ × 2.5 + [mg/L magnesium] ____ × 4.1 = ☐ mg/L as $CaCO_3$

TOTAL DISSOLVED SOLIDS: (CALCULATED)

TDS = [Total Cations] ____ + [Total Anions] ____ = ☐ mg/L

METHOD 02

METHOD for DENSITY of LIQUIDS

A) REFERENCES:

A.S.T.M. D4052

Instrument Manual - Calculating Digital Densitometer DMA 45

Density and Related Values

B) SCOPE:

This method can be used on any liquid which meets the following criteria:

1) Has a density less than 2.0 g/cm³.
2) Remains liquid at the test temperature.
3) Has a viscosity, at the test temperature, low enough to facilitate filling and emptying the sample cell.
4) Is not a strong caustic.

The temperature limits of the densitometer (Note #1) is the other restriction to the use of this method.

5) The temperature of the measurement is within the limits of the instrument. (Note #1)

C) SUMMARY:

The liquid sample is injected into an oscillating tube, resulting in a change in the period of the oscillation. This change is detected and the density of the sample is automatically calculated from the calibration factors and displayed to four decimal places as g/cm³.

D) INTERFERENCES:

Air or vapour bubbles will cause low or erratic results. Care must be taken in sampling and filling the cell to avoid incorporation of air into the sample.

E) SAFETY:

1) Acetone and Xylene, used in cleaning the cell, as well as hydrocarbon samples are highly flammable. Care should be exercised to maintain adequate ventilation of fumes and to prevent contact with sources of ignition.

2) Normal petroleum laboratory procedures.

3) Refer to the Material Safety Data Sheets for detailed information on reagents.

F) APPARATUS:

1) Densitometer, DMA 45
2) Temperature Bath, Neslab RTE-8
3) Syringes, 3 cc Luer tip
4) Syringe Adaptor
5) Aspirator
6) 1 L Separatory Funnel
7) 2-Winchester Bottles for cleaning solvents
8) 1/8" I.D. Tygon tubing
9) ¼" Vacuum tubing
10) Warm Water Bath

G) REAGENTS:

1) Xylene, Technical Grade
2) Acetone, Technical Grade

H) PROCEDURE:

a) Calibration:

The densitometer should be calibrated at least once per week. A calibration should also be made if it is suspected that erroneous readings are being generated.

Calibration may be monitored on a daily basis by checking a sample of distilled water and comparing the reading to that listed in the tables. (record reading in your laboratory notes.)

1) Bring the system to temperature (t) of interest and allow the instrument to stabilize (approximately 10 minutes).

2) The cover plate on top of the instrument is removed to expose the display selector switch and the constant buffer switches.

3) Set the display selector switch to position "T" using a small screwdriver.

4) Switch light on to view the sample compartment.

5) Using a syringe, introduce distilled deionized water into the cell until full. (Do not remove the syringe).

6) Switch the light off and allow the cell to come to temperature equilibrium. (Reading is repeated at least 10 times).

7) When a stable reading is obtained, record the reading as T_{H_2O} (period of water). (Note #2)

8) Wash the water sample out of the cell using acetone and blow dry (refer to cleaning procedures).

9) Once the cell is dry, the air pump and light are switched off and the air filled cell is allowed to equilibriate.

10) Record the reading as T_{air} (period of air).

11) Read the barometric pressure (P) in mm of Mercury. (See Method L1)

12) Obtain the water density (D_{H_2O}) at operating temperature from the tables in the Technical Information section.

13) Calculate the air density (D_{air}) at the operating temperature and the instrument constants A and B using the following equations.

$$D_{air} = \frac{0.0012930}{1 + 0.00367*t} * \frac{P}{760}$$

$$A = \frac{T_{H_2O} - T_{air}^2}{D_{H_2O} - D_{air}}$$

$$B = T_{air}^2 - A*D_{air}$$

14) Update the constants entered on the reverse side of the switch cover panel (pencil only) and the calibration record in the back of the instrument manual.

15) Set the buffer switches for the constants A and B to reflect the new values.

16) Set the display selector switch to (density).

17) Replace the cover plate.

b) Analysis:

1) Turn on the sample compartment light and ensure the cell is empty and clean.

2) Load a disposable 3 ml plastic syringe with sample (Notes # 3 & 4).

3) Carefully inject the sample into the cell through one of the sample ports (usually the upper) until the narrow portion of the U tube is completely filled. Leave the syringe attached to the instrument. (Note #5)

4) Turn off the sample compartment light and allow the sample to come to temperature equilibrium (2-3 minutes). Equilibrium has been reached when a stable reading has been achieved (i.e. the instrument repeats the same value 10 times).

5) Record the density reading in your laboratory notes as Reading #1.

6) Inject an additional 1.0 ml of the sample into the cell and obtain a second reading. Record the stable reading as Reading #2 in your laboratory notes.

I) CLEANING:

1) Attach the aspirator vacuum source to one of the ports of the cell.

2) Allow the sample to be suctioned from the cell.

3) Attach the hose of the appropriate cleaning solvent to the other port of the cell.

For oil samples, use Xylene.
For water samples, use distilled water.

4) When the cell has been flushed sufficiently to remove all of the sample, remove the cleaning solvent hose and rinse with acetone to remove the last traces of solvent.

5) Remove the acetone hose and air dry the cell by connecting and activating the built-in air pump on the densitometer.

6) After the cell is dried (1-2 minutes) the air pump can be disconnected and turned off. The cell is now ready for the next sample.

7) After extended use, deposits will accummulate on the tube surfaces. These can be removed by washing with chromic acid overnight. Following this, a recalibration is required.

8) Clean the exterior of the instrument and the surrounding work area as required.

J) **CALCULATIONS**: Density (Kg/m^3) =

$$\text{Density } (g/cm^3) \times \frac{1{,}000{,}000\ cm^3}{m^3} \times \frac{1\ Kg}{1000\ g}$$

K) **PRECISION**: Duplicate readings shall not differ by more than ± 0.0002 g/cm^3 (± 0.2 kg/m^3).

L) **QUALITY CONTROL**: 02.4

1) If the difference between duplicates is greater than 0.0002 g/cm^3, inject an additional 1.0 ml of sample into the cell and obtain a third reading.

2) Report the higher of the two readings that have a difference of less than 0.0002 g/cm^3.

3) If the difference is still greater than 0.0002 g/cm^3, clean the cell and repeat the analysis.

M) **REPORTING**:

a) Significant Figures: (Note #6)

1) The higher value of duplicate readings shall be reported as the density of the sample.

2) Results should be reported to four (4) significant figures whether reported as g/cm^3 or kg/m^3.

b) **Format:**

Results shall be reported as follows:

Density @ ##.# °C = 0.#### g/cm³ or ###.# kg/m³

i.e. Density @ 15.0 °C = 0.9478 g/cm³ or 947.8 kg/m³

N) NOTES:

1) The densitometer has a temperature range of 0°C to 50°C. The upper temperature limit of the bath has been set at approximately 45°C to prevent accidental overheating of the densitometer. If a temperature lower than 8°C is desired the bath fluid (deionized water) will need to be replaced by a water/glycol mixture to prevent freezing.

2) To test the temperature control stability, monitor the T_{H_2O} for 15 minutes. The value should not vary more than 1 unit in the last significant figure.

3) When handling heavy viscous crude oils, warm the sample (50-60°C) before loading the syringe. This minimizes the entrainment of gas bubbles in the sample which affects the accuracy of the density determination.

4) When working with lighter crudes, the syringe can be loaded at ambient temperatures but should still be allowed to stand tip down to allow gas bubbles to collect at the plunger.

5) Viscous samples should be injected slowly and with care to minimize bubble formation and to prevent cell damage from over pressuring.

6) Significant figures - see Technical Information section.

REVISION DATE: 1988 DECEMBER 15

METHOD W-10

ANALYSIS OF ANION CONTENT IN WATER

A) **REFERENCES:**

1) ASTM D4327-84 Anions in Water by Ion Chromatography
2) EPA The Determination of Inorganic Anions in Water by Ion Chromatograph - Method 300.0
3) Dionex Ion Chromatography Cookbook

B) **SCOPE:**

This method is used in the analysis of various types of waters to separate and quantitatively detect chlorides, fluorides, nitrates, phosphates and sulphates, using the Dionex 2000i ion chromatograph. (Note #4)

C) **SUMMARY:**

The sample is injected into the sample port, where it passes through a prefilter (bed support) which removes any undissolved particles from getting on the chromatographic columns. The sample is then pushed by the eluent (aqueous $NaHCO_3$ & Na_2CO_3) (onto the neutral guard column) where unwanted organic molecules are removed. The next column that the sample travels through is an anion guard column which removes any large anions that may contaminate the actual separator column. (The neutral guard column and the anion guard column are expendable and can easily be replaced while the actual separator column is much more expensive to replace.) The partially separated sample then enters the separator column where the majority of anion separation occurs. Because each anion has a different affinity for the ion exchange resin sites it passes some anions stay in the stationary phase. longer than others before being displaced by the eluent. This produces bands of the same anion travelling together along the column. After the separator column the anions and eluent continue along to the suppressor, where the eluent exchanges its Na+ for H+. This increases the hydronium ion content forcing the equilibrium to favour H_2CO_3. Carbonic acid has a much lower conductivity than its individual ions ($H+H+CO_3^=$). This reduces the background noise (conductivity) so that the anion to be detected is the dominant ionic species. The anions are detected by a conductivity cell. As the conductivity created by each anion band is measured, a peak is produced by the conductivity versus time plot. The concentration of each anion present is determined by peak height or area. The solution leaves the conductivity cell and enters a waste reservoir, and is then discarded.

D) **INTERFERENCES:**

1) The suppressor must be working to its full potential for ion exchange at all times. If it is not, the background noise (conductivity) will be higher than the anion conductivities. (i.e. peaks will be covered by baseline and never be seen or detected.)
2) The glassware used to make solutions, eluents and regenerant must be clean, along with clean syringes to prevent contamination from occurring. (See Cleaning Procedure for Trace Analysis)
3) If there are other ions in the sample, with the same retention times they may overlap with those anions being analyzed and cause false readings(i.e. organic acids (formate & acetate) elute at the same time as fluoride but have a much broader peak.
4) As the columns are used repeatedly over time, they begin to deteriorate due to contaminants building up on the ion exchange resin sites. This reduces the efficiency of your columns and therefore your analysis.
5) Air bubbles in the tubing, columns, pump, or conductivity cell will cause baseline upset.
6) Water dip occurs very close to F & Ce peaks - add concentrated eluent to sample and insure proper integration at peak shear water dip.

E) **SAFETY:**

1) Use normal laboratory safety procedures.
2) Rubber gloves, safety glasses and protective clothing must be worn when handling H_2SO_4.
3) Refer to Material Safety Data Sheets for detailed information on reagents.

F) **APPARATUS:**

1) Ion Chromatograph with a temperature compensated conductivity meter and a constant flow pump 2 - 5 ml/min.
2) A neutral guard column to remove unwanted organic molecules (NG1).
3) A separator guard column to protect separator column (AG4A).
4) A separator column to separate anions (AS4A).
5) A suppressor column to exchange sodium for hydrogen ions (AMMS).
6) Eluent and regenerant reservoirs.
7) A recorder, preferrably with an integrator.
8) Grade A volumetric flasks, and pipettes for dilutions.
9) Syringes.

G) REAGENTS:

1) Anion Regenerant: An 18 millimolar H_2SO_4 solution is required, add 2 ml of concentrated (36N) H_2SO_4 to four (4) litres of deionized water that has a resistance greater than 18 mega ohms. The absolute accuracy of the regenerant solution is not critical. A roughly calibrated plastic jug is sufficient for solution preparation.
2) Mixed Anion Eluent: A 1.8 millimolar (0.763 g/4L) Na_2CO_3 and 1.7 millimolar (0.571 g/4L) $NaHCO_3$ aqueous solution is required. All reagents used for the eluents must be high purity. The deionized water must also have a resistance greater than 18 mega ohms, and must be degassed by purging with helium for 10 minutes.
3) Anion Standards - (Stock Solutions): A 1000 mg/L stock solution of each anion (fluoride, chloride, phosphate, nitrate and sulphate) is made from high purity reagents that have been dried at 105°C and cooled in a dessicator. The standards are made by serial dilutions using the stock solutions and 18 mega ohm deionized water, ranging from detection limits to 10 mg/L. Weights must be corrected for reagent purity.

   F^- from NaF = 2.2100 g/L
   $PO_4 = NaH_2PO_4 \cdot 2H_2O$ = 1.64273 g/L
   Cl^- from $NaCl$ = 1.6485 g/L
   NO_3^- from $NaNO_3$ = 1.3708 g/L
   $SO_4 = Na_2SO_4$

H) PROCEDURE:

a) Start Up:

If on Cation Mode:

1) Start pump and begin flushing deionized water through the system for 5 minutes at 2 ml/m.
2) Shut off pump and switch over to Anions.
3) Turn on Anion eluent and continue start-up procedure.

If on Anion Mode:

1) The ion chromatograph should be assembled according to manufacturer's specifications.
2) Start pump and begin flushing eluent through the system (2 ml/min.).
3) Turn regenerant on so it has a flow of 2-3 ml/min.
4) Turn compressed air and conductivity meter on.
5) Set conductivity on a 30 microSeimen range.
6) The instrument should be left for about one (1) hour to pump eluent through the columns, suppressor and detector until the pressure and baseline has stabilized.
7) Once the instrument has been allowed to stabilize, turn the auto-offset on.
8) Run blank to check baseline and for contamination.

b) Calibration:

1) Inject 2-3 mls of each standard in increasing anion concentration.
2) Do not inject next sample until the previous sample has been analyzed. If the sample is placed in the injection port before analysis, and there is a leak in one of the valves, the sample will be lost or diluted.
3) For each injection, peak height or area for each anion is recorded.
4) Make a graph of peak height, or area versus concentration of the anion. The graph will produce a line that will be used in determining the concentration of each anion of interest for each sample that is to be analyzed.

c) Analysis:

1) Once the calibration procedure is complete, the samples are now to be analyzed.
2) Prepare sample table with dilution valves.
3) Analyze.
4) Once each sample has been analyzed, go to the standards graph and determine the concentration of each anion of interest in the samples if integrator is not working.

d) Shutdown:

1) Shut off pump, eluent, regenerant, conductivity cell, auto-offset and compressed air.
2) Empty waste from the waste reservoir.

I) CLEANING:

1) See Cleaning Procedure for Trace Ion Analysis.
2) All glassware and syringes must be cleaned with 1N NaOH, then washed three (3) times with deionized water and soaked in deionized water overnight and washed three (3) more times in deionized water.
3) See maintenance manual for I.C. and column cleaning.

J) CALCULATIONS: Not Applicable.

K) PRECISION:

1) Tests were run on a single-operator in which one (1) standard was run ten (10) times to determine the accuracy and precision of the Dionex 2000i ion chromatograph.

Standard 95% Relative

Anion	Standard (mg/L)	Mean (mg/L)	Standard Deviation (SD)	95% Confidence Level	Relative Standard Deviation (%,RSD)	Detection Limits mg/L
Fluoride	1.0	0.9857	0.00947	± 0.02	0.96	0.1
Chloride	2.0	2.0142	0.02015	± 0.05	1.00	0.1
Nitrates	3.0	2.9983	0.01614	± 0.04	0.54	0.2
Phosphates	3.0	2.9914	0.01641	± 0.04	0.55	0.1
Sulphates	3.0	3.0028	0.01707	± 0.04	0.57	0.2

Detection limits can be lowered by lowering microSiemen range to 3 µS and lowering standard concentrations.

L) QUALITY CONTROL:

1) Conductivity meter must be calibrated after every cleaning.
2) The anion calibration standards should be checked against each other and by using a certified standard monthly. (Note #5) The anion concentrations inthe standards may change due to evaporation; or contamination. The lids must always be kept on the standards to decrease the possibility of evaporation.
3) Deionized water must have a resistance greater than 18 mega ohms to be used for all standards, eluents, regenerant and dilutions.
4) The deionized water must be degassed by purging it with helium when mixing up the eluents.
5) All reagents used must be of high purity.

M) REPORTING:

a) Significant Figures:

1) Two (2) significant figures are reported.
2) When dealing with trace amounts of an anion, the Dionex 2000i is accurate to one (1) decimal point.

b) Format:

1) All results from the ion chromatograph are listed as mg/L.

N) NOTES:

1) The water is the first thing to come off the column and be detected. Water has a much lower conductivity than that of the eluent. A dip is made on the recorder paper when the water is detected by the condictivity meter. This sometimes causes integration problems if you have an integrator attached to your recorder. The water dip may be eliminated by adding concentrated eluent to all standards and samples.

2) To check if glassware is indeed clean after the cleaning procedure, place deionized water (with an 18 mega ohm resistance) in the container and let stand for a few minutes. Inject deionized water from glassware into the ion chromatograph. The ion chromatograph will detect if any contaminants are still present on the glassware.

3) The hands and fingers are the worst form of contamination to occur. Keep hands away from syringe tips, and hands out of standard and sample solutions.

4) This method can also be used for nitrites, sulfites, and thiosulfites but due to the instability of these products a reference standard is used.

   i.e. Nitrate for Nitrite
   Sulfate for Sulfite and Thiosulfite

5) Results should be within precision of the method (1% RSD).

REVISION DATE:

METHOD W13

pH OF WATER

A) REFERENCES:

1) Alberta Environment - Methods Manual for Chemical Analysis of Water and Wastes - METHOD NAQUADAT No. 1030IL
2) Operating Instructions PHM82 Standard pH Meter Radiometer

B) SCOPE:

This method is used for determining the pH of all types of water samples with pH 0 - 14.

C) SUMMARY:

Two electrodes, a glass electrode and a reference electrode (usually a saturated calomel) are used for pH measurement. The glass electrode has a glass membran which forms a partition between the liquid sample and an internal solution of constant pH. The potential developed between the glass electrode and the saturated calomel electrode is measured electrometrically.

pH is defined as the logarithm of the reciprocal of the hydrogen ion concentration (the hydrogen ion activity) in moles per liter.

D) INTERFERENCES:

1) High sodium concentrations at a pH above 10 corrections can be made according to a chart supplied by electrode manufacturer.
2) Oil and solids interfere with efficiency of probe - clean sample as much as possible.

E) SAFETY:

1) Routine safety equipment use is all that is required (eg. safety glasses, plastic gloves).

F) APPARATUS:

1) pH meter - PHM82 Radiometer
2) Glass electrode
3) Reference electrode (saturated calomel)
4) Stirring plate
5) Stirring bar
6) Beakers (100 ml)

G) **REAGENTS:**
1) Commercial standard pH buffer solutions - pH 4.0, 7.0, 10.0.
2) Saturated KCl solution.
3) Deionized water.

H) **PROCEDURE:** (Note #1)

a) CALIBRATION:
1) Set temperature compensator of the pH meter to room temperature.
2) Place pH 7.0 buffer solution in a beaker with a stirring rod, place on stirring plate and set sample stirring.
3) Place the electrodes in the solution being careful not to let the electrodes get hit with the stir bar.
4) Press the hold button off (light is off) and allow the pH to stabilize.
5) Use a combination of the sensitivity button and the buffer knob to adjust the pH reading to 7.00. Allow to stabilize.
6) To change the sample, press the hold button, turn off the stirrer and remove the electrodes from the solution.
7) Wash the electrodes with deionized water and dry with a soft tissue.
8) With the 4.00 or the 10.00 pH buffer solution repeat steps 2 to 7. (Note #2)
9) As a check determine the pH 7.0 buffer solution.

b) ANALYSIS:
1) **Samples are to be kept cool and analyzed as soon as possible.**
2) Place the water sample to evaluated in a beaker or paper cup together with a stirring bar and place on the stirring plate. Set the sample stirring.
3) Place the electrodes in the sample solution being careful not to let the electrodes get hit with the stir bar.
4) Press the hold button off (light is off) and allow the pH to stabilize.
5) Record the number observed.
6) Press the hold button on, turn off the stirrer and remove the electrodes from the sample.
7) Wash the electrodes with deionized water.
8) When the analyses are finished, clean the electrodes as per the cleaning procedure.
9) Store the calomel electrode in a pH 7.00 buffer solution. Store the glass electrode in a 4.00 to 7.00 ph buffer.

I) **CLEANING:**
1) Electrodes:
   a) CALOMEL ELECTRODE:
      i) Soak electrode in xylene or chloroform for 15 minutes.

   - ii) Rinse electrode with isopropyl alcohol or acetone.
   - iii) Push KCl electrolyte into the top of the electrode and watch for drops to form out of the bottom porous plug.
   - iv) If the porous plug is still dirty or you cannot get good readings when you try to calibrate, repeat steps i to iii.
   - v) If this still does not work soak in 1N HCl for 15 minutes, rinse with distilled water and squeeze a drop of KCl through the porous plug.
   - vi) If this still does not work soak in chromic acid for a maximum of 15 minutes, rinse with distilled water and squeeze a drop of KCl through the porous plug.

b) GLASS ELECTRODE:
   - i) Soak electrode in xylene or chloroform for 15 minutes.
   - ii) Rinse electrode with isopropyl alcohol or acetone.
   - iii) If the electrode still does not appear clean, soak in 1N HCl for up to 24 hours and rinse with distilled water.
   - iv) If the electrode is still not clean, soak for a maximum of 15 minutes in chromic acid and rinse with distilled water.

c) HACH ELECTRODE:
   - i) To clean, when electrode response becomes sluggish, immerse the electrode tip in 0.1N hydrochloric acid for 2 minutes, followed by immersing in 0.1N sodium hydroxide for 2 minutes, then immersion in 0.1N hydrochloric acid for another two minutes. Rinse with deionized water and soak in deionized water for at least 15 minutes.
   - ii) This electrode can be stored air-dry. It responds well when re-immersed in strong buffer.
   - iii) The electrolyte solution in the reference electrode (2.2N potassium chloride) may form crystals. This can be corrected by immersing the electrode in warm water (30°C).

d) All beakers and other glassware are to be washed with soap and water and rinsed with distilled water.

J) **CALCULATIONS:** 1) None.

K) **PRECISION:** 1) The meter is accurate to ±0.10 pH.

L) QUALITY CONTROL:

1) Analyze samples with known pH to check the calibration and accuracy of the instrument.

M) REPORTING:

a) Significant Figures:

1) Report to the second figure after the decimal.

b) Format:

1) Report as:

pH X.XX

N) NOTES:

1) The outlined method is for the electrode system designed by Radiometer. We also have an electrode system designed by Hach which consists of a dispensette for electrolyte solution and plastic electrode. This system only works for clean waters.
2) It is only necessary to use two buffer solutions to set the calibration. Whether you use 4.00 or 10.00 as your second, buffer is determined by the pH of the actual samples.

REVISION DATE:

METHOD W14

ALKALINITY

A) **REFERENCES:**

1) Standard Methods for the Examination of Water and Wastewater. Method 403

B) **SCOPE:**

This method is used to determine the alkalinity of all field waters requiring stiff patterns, refinery cooling water, and any waters where you are interested in the scaling tendencies.

C) **SUMMARY:**

A sample of water containing phenolphthalein indicator is titrated with N/50 H_2SO_4 to an endpoint (where the colour changes from pink to clear) to obtain the carbonate alkalinity. Methyl orange indicator is then added and the titration is continued to an endpoint (where the colour changes from a yellow to a pink) to obtain the total alkalinity. The carbonate, bicarbonate and hydroxide hardness is then calculated.

Alkalinity is defined as the quantitative capacity of a water to neutralize a strong acid to a designated pH.

D) **INTERFERENCES:**

E) **SAFETY:**

1) Refer to MSDS sheets for information on sulfuric acid

2) Use of basic laboratory safety is required (safety glasses, gloves).

F) **APPARATUS:**

1) 25 ml titration buret
2) Bottle to hold titrant
3) Ceramic dish (100 ml capacity)
4) Glass rod with rubber policeman
5) Pipette (suitable)

G) **REAGENTS:**

1) N/50 sulfuric acid - reagent (stock from Betz)
2) Phenolphthalein Indicator - dissolve 5 g of phenolphthalein disodium salt in deionized water and dilute to 1 liter.
3) Methyl Orange Indicator - dissolve 500 mg methyl orange powder in deionized water and dilute to 1 liter.

H) **PROCEDURE:**

a) CALIBRATION: None.

b) ANALYSIS:

1) Pipette a suitable quantity of the water to be tested (25 ml is suggested) into the ceramic dish.
2) Add 25 ml of deionized water.
3) Fill the titration buret to the zero mark with titrant.
4) Add 3 or 4 drops of phenolphthalein indicator to the ceramic dish.
5) If the water turns pink, titrate with N/50 sulfuric acid until the water just turns clear. Record the volume and call it P.
6) If the water stays clear, record P as 0.
7) Add 3 or 4 drops of methyl orange to the sample. The colour will be yellow if carbonates are present and an orange-pink if no carbonates are present. Continue the titration until an orange-pink colour appears. (Note #1) Record the volume and call it T. Record T as 0 if no carbonates are present.

I) **CLEANING:**

1) Wash the ceramic dish and the stir stick with deionized water before reusing.

J) **CALCULATIONS:**

1) The data from the titrations is to be interpreted as follows:

TITRATION RESULT	HYDROXIDE ALKALINITY As $CaCO_3$	CARBONATE ALKALINITY AS $CaCO_3$	BICARBONATE ALKALINITY As $CaCO_3$
P = 0	0	0	T
P < 1/2T	0	2P	T - 2P
P = 1/2T	0	2P	0
P > 1/2T	2P - T	2(T - P)	0
P = T	T	0	0

Factors to convert $CaCO_3$ to:

mg/L OH = Hydroxide = $CaCO_3$ x 0.340

mg/L CO_3 = Carbonate = $CaCO_3$ x 0.600

mg/L HCO_3 = Bicarbonate = $CaCO_3$ x 1.219

K) **PRECISION:** 1) The precision of the analyses is difficult to establish because of the variation in sample characteristics.

L) **QUALITY CONTROL:**

1) Analyze a sample of known alkalinity.

M) **REPORTING:**

a) **Significant Figures:** (See Technical Section)

1) Report to three significant figures.

b) **Format:** 1) Report as:

Alkalinity, mg/L	XXX
eg. Carbonates, mg/L	215

N) **NOTES:** 1) Do not refill titration buret.

REVISION DATE:

METHOD W15

METALS BY ATOMIC ABSORPTION

A) **REFERENCES:** 1) Analytical Methods for Atomic Absorption Spectrophotometry, Perkin Elmer

B) **SCOPE:** This method is applicable to the analysis of surface, waste, and produced waters and any materials which can be dissolved or converted by chemical processes to aqueous solutions where the metal contents are in the mg/L range.

C) **SUMMARY:** An aqueous solution of a metal is aspirated into a burner fired by either an acetylene-air or acetylene-nitrous oxide mixture. A light beam, from a hollow cathode lamp of the metal being analyzed, is passed through the flame. The absorbance of the beam caused by the metal in the flame is detected and compared against a standard curve to yield the concentration.

D) **INTERFERENCES:**

1) Some metals are readily excited to emission which means the atoms cannot absorb light. Sodium chloride is added to such metal solutions to reduce this interference.
2) Some metals react strongly with other ions or with the oxygen in the flame to form very stable compounds or refractory oxides. These will not allow the metal to atomize in the flame. This effect is countered by adding lanthanum chloride to the solutions or by using the hotter acetylene-nitrous oxide flame.
3) Specific chemical interferences for each metal are described in the Standard Conditions Table, Table 1 (see Standard Conditions Section of the Operator's Manual for more details) along with techniques for handling each one.
4) Oil or sediment can block and foul aspirator tubes and nozzles. This is avoided by filtering the samples.

5) Flame absorbances and emissions may interfere with the absorption bands of some elements. Using an alternate wave length will avoid this situation.
6) Self-absorption causes non-linearity and usually occurs at high concentrations. Dilution of these strong solutions usually corrects this problem.
7) Matrix interferences result from the physical characteristics of the solvent (viscosity, high surface tension, combustible solvent). To overcome these problems the operator should use the same solvent in standards and samples. Dilute high TDS samples or use the method of standard addition where all else fails. (Note #1)
8) Background interferences caused by light scattering of particles or molecular absorbance can be avoided by using a hotter flame or by using the Centurium Arc Background Correction.

E) SAFETY:

1) Acetylene:
   a) Cylinders transported on their side should be stored upright for at least 24 hours before using. Failure to do so may cause acetone/acetylene to be drawn into the regulator and result in a violent explosion.
   b) The main cylinder valve should never be opened more than 1/3 to 1/2 turn to prevent acetone excaping from the cylinder during a catastrophic line failure. It also permits quicker shutdowns under these circumstances.
   c) Acetylene cylinders should never be drawn below 520 kpa (75 psi).
   d) The acetylene delivery pressure to the A.A. should never exceed 70 kpa (10 psi) as pure acetylene spontaneously dissociates at high pressure.
   e) For more information, see Appendix A.
2) When handling hot burner heads, insulated gloves should be used to prevent burns.
3) Before using the A.A., ensure the trap on the drain line is filled with water to prevent escape of gases through the drain container.
4) Ventilation fan must be used to remove toxic combustion gases or vented fuel gases.
5) Refer to the Material Safety Data Sheets for detailed information on reagents and fuel gases.

F) APPARATUS:

1) 3 - 10 ml dispensettes with 1 litre reservoir bottles.
2) 100 ml volumetric flasks (as required).
3) 500 ml volumetric flasks (as required).

4) 500 ml plastic bottles with caps (as required).

5) Selection of pipettes suitable for making accurate dilution.

6) Perkin-Elmer 3030 Atomic Absorption Spectrophotometer.

G) **REAGENTS**:

1) Concentrated nitric acid
2) Sodium chloride
3) Lanthanum chloride
4) Acetylene
5) Nitrous oxide
6) 1000 mg/L standard solutions

H) **PROCEDURE**:

a) **CALIBRATION**:

1) Prepare 3 calibration standard and 1 reference standard as per Table #2. (Note #2)
2) Turn on ventilation fan and instrument power.
3) Install the required burner head.
4) Open the main acetylene valve 1/3 to 1/2 turn.
5) Open the air valve and nitrous oxide valve (if required).
6) Set the appropriate fuel and oxidizer flow rates:

	FUEL	OXIDANT
Air/Acetylene	20	45
N_2O/Acetylene	33	33

7) Install or rotate the appropriate hollow cathode lamp into position.
8) Recall the appropriate program from the User Index. (Note #3)
9) Set the lamp current to the continuous operating valve stamped on the lamp.
10) Set the slit-width and wavelength as per the method on the monitor.
11) Set the instrument on Set-up mode, optimize the wavelength and lamp position (see Operator's Manual).
12) Light the burner and allow a minimum of 15 minutes for warm-up.
13) Aspirate the Sensitivity Check Standard and optimize the burner vertical, horizontal, and rotational controls and optimize the aspirator flowrate (see Operator's Manual).
14) Switch to an operating mode, aspirate a blank and set zero.
15) Starting with the lowest concentration, aspirate each of the calibration standards and press its calibration key. Re-zero between each standard.
16) Check the calibration by reading the reference standard. This should agree to within 4.0% of its known concentration.

b) ANALYSIS:

1) All samples, except silica (Note #4) should be preserved by acidifying with 1.0% by volume of concentrated nitric acid.
2) Re-zero the instrument using a blank.
3) Add any acids and reagents as listed in the Standard Conditions (Table 1) for the element in question.
4) Samples with oil or suspended solids require filtration before analysis.
5) Aspirate the sample and read the concentration from the monitor.
6) If the sample is too concentrated, make an accurate dilution using pipettes and volumetrics, re-treat with the appropriate reagents and repeat the analysis.
7) If the concentration is too low, the flow spoiler may be replaced with the impact bead for air/acetylene flames only. (Note #5)
8) After every 5 samples, run a calibration standard to ensure accuracy of calibration.

I) CLEANING:

1) Glassware:
   a) Normal laboratory procedure should be adequate.

2) Aspirator (Minor Cleaning):
   a) If the aspirator becomes clogged with oil or sediments it can frequently be cleared by passing a wire through the aspirator tubing and assembly. (Note #5)
   b) Re-check the calibration before continuing with analysis.

3) Aspirator (Major Cleaning):
   a) If the above fails to clear the problem, shut down the burner and dismantle the aspirator assembly.
   b) Wash the metal parts and tubing with solvent and/or soap and water.
   c) Use the cleaning wire to clear any debris from the tubing or needle.
   d) Re-assemble the aspirator (Figure #1).
   e) Re-light and re-warm the burner, adjust the aspirator and recalibrate before continuing the analysis.

4) Burner Heads:
   a) All burner cleaning requires shutting down of the burner. (Note #7).
   b) Carbon, salt and other deposits on the burner slot can be removed using the razor blade or scraper tool provided.
   c) More tenacious deposits can be removed by soaking the head in water and brushing with a bottle

brush and using the scrapers.

d) Burner heads are to be cleaned at the completion of each sample batch.

5) Premix Chamber:

a) After completion of each batch of samples the premix chamber should be flushed with distilled water to remove salt solutions and deposits.

b) If the walls are oily, the burner assembly should be dismantled, solvent washed, wiped clean and water rinsed. The unit is then re-assembled. (Figure #2)

J) **CALCULATIONS:** 1) $C = \frac{V \times A}{S}$

V = Final dilution volume, ml
S = Sample aliquot, ml
A = Instrument reading, mg/L
C = Original sample concentration, mg/L

K) **PRECISION:** 1) Duplicate results shall agree to within 5.0 % of each other.

L) **QUALITY CONTROL:**

1) Results which fail the precision criterion shall be repeated.
2) A calibration check shall be run after every fifth sample, utilizing the standard closest to the last sample analyzed.
3) Failure of the standard to fall within 4.0 % of its known value requires the operator to recalibrate and repeat the five previous analyses with another calibration check included.

M) **REPORTING:**

a) Significant Figures: (See Technical Section)

1) All results shall be reported to 3 significant figures.

b) Format: 1) The following format is recommended:

Metal, mg/L	XX.X
eg. Calcium, mg/L	1 230.

N) **NOTES:** 1) See the Operator's Manual for the method of standard additions. This approach is rarely used at this time and therefore does not warrant being published and is only included for completeness.

2) One of the four standards doubles as the Sensitivity Check.
3) See the Operator's Manual for selecting and initializing new user index programs.
4) Silica samples should never be acidized. Cool to 4.0°C and analyze within 7 days.
5) The use of the impact bead doubles the sensitivity of the element with air/acetylene flames. Use of the bead with N_2O/acetylene flames usually has no effect and therefore is not recommended.
6) This can be done while the instrument is operating, avoiding a shut-down and re-warming of the burner.
7) Exercise care in handling hot burners. Use insulated gloves and avoid contact with hot components in the burner compartment.

REVISION DATE:

APPENDIX A

SAFETY GRAM

FROM: SASKATCHEWAN HIGHWAYS AND TRANSPORTATION

IMPROPER STORAGE OF ACETYLENE CYLINDERS CAN LEAD TO EXPLOSION

An acetylene cylinder lying on its side can lead to an explosion caused by the withdrawal of acetone. Acetylene cylinders must stand upright.

Acetylene cylinders are made of steel, and are packed with a monolithic filler to occupy all internal empty space. Acetone is pumped into the cylinder; then with special equipment, acetylene is pumped into the cylinder where it is immediately absorbed into the acetone. This causes the liquid to expand and to occupy practically all the pores of the filler material.

Acetylene gas cannot be safely used or stored at a pressure greater than 25 lb per sq. in. or where the empty space volume is greater than 1/8 cu. in.

When the cylinder valve is opened, a pressure drop occurs and the acetone gives up the acetylene. If the rate of withdrawal is greater than 1/7 of its volume per hour, acetone will be drawn out with the acetylene, leaving empty spaces where free acetylene can collect under high pressure. (This is the reason for not opening the valve more than one full turn.) Of all fuel gases used commercially, acetylene is one of the more dangerous because it disassociates with explosive violence when subjected to high pressure or temperature.

Acetone can also be drawn out if a cylinder is used within two hours after it has been lying on its side. Therefore, acetylene cylinders must be stored in an upright position.

--Les Longbrook
Al Cohen Construction Co.
Denver, Colo.

NOTE: Our Department recommendations are that if acetylene cylinders were transported in a horizontal position they should be upright for at least 24 hours before use, in order to ensure the acetone is not drawn into the guages etc.

The acetylene cylinder should not be opened beyond 1/3 to 1/2 turn for use, this precaution can be beneficial in an emergency situation where quick shut down is required.

Safety Section

TABLE #1

STANDARD CONDITIONS FOR ATOMIC ABSORPTION

ELEMENT	λ (nm)	SBW (nm)	FLAME GASES	SENS. CHECK(a)
Ag	328.1	0.7	A-Ac	2.5 (e)
Al	309.3	0.7	N-Ac	50.0 (b)
As	193.7	0.7	A-Ac	45.0 (e)
Au	242.8	0.7	A-Ac	15.0 (e)
B	249.7	0.7	N-Ac	600.0
Ba	553.6	0.4	N-Ac	20.0 (b)
Be	234.9	0.7	N-Ac	1.5
Bi	223.1	0.2	A-Ac	20.0 (e)
Ca	422.7	0.7	A-Ac	4.0
Cd	228.8	0.7	A-Ac	1.5 (e)
Co	240.7	0.2	A-Ac	7.0 (e)
Cr	357.9	0.7	A-Ac	4.0 (e)
Cs	852.1	1.4	A-Ac	10.0 (b,e)
Cu	324.8	0.7	A-Ac	4.0 (e)
Dy	404.6	0.2	N-Ac	45.0 (b)
Er	400.8	0.2	N-Ac	30.0 (b)
Eu	459.4	0.4	N-Ac	30.0 (b)
Fe	248.3	0.2	A-Ac	5.0 (e)
Ga	287.4	0.7	N-Ac	60.0 (b)
Gd	368.4	0.2	N-Ac	850.0 (b)
Ge	265.1	0.2	N-Ac	100.0
Hf	286.6	0.2	N-Ac	450.0 (c)
Hg	253.7	0.7	A-Ac	200.0 (e)
Ho	410.4	0.2	N-Ac	40.0 (b)
In	303.9	0.7	A-Ac	35.0 (e)
Ir	264.0	0.2	A-Ac	500.0 (e)
K	766.5	1.4	A-Ac	2.0 (b,e)
La	550.0	0.4	N-Ac	2500.0 (b)
Li	670.8	1.4	A-Ac	2.0 (e)
Lu	336.0	0.2	N-Ac	250.0 (b)
Mg	285.2	0.7	A-Ac	0.3 (e)
Mn	279.5	0.2	A-Ac	2.5 (e)
Mo	313.3	0.7	N-Ac	30.0
Na	589.0	0.4	A-Ac	0.5 (b,e)
Nb	334.4	0.2	N-Ac	700.0 (c)
Nd	492.4	0.4	N-Ac	350.0 (b)
Ni	232.0	0.2	A-Ac	7.0 (e)
Os	290.9	0.2	N-Ac	45.0
P	213.6	0.2	N-Ac	14000.0
Pb	283.3	0.7	A-Ac	20.0 (e)
Pd	244.8	0.2	A-Ac	10.0 (e)
Pr	495.1	0.4	N-Ac	2000.0 (b)
Pt	265.9	0.7	A-Ac	100.0 (e)
Rb	780.0	1.4	A-Ac	30.0 (b,e)
Re	346.0	0.2	N-Ac	650.0
Rh	343.5	0.2	A-Ac	9.0 (e)
Ru	349.9	0.2	A-Ac	30.0 (d)
Sb	217.6	0.2	A-Ac	25.0 (e)
Sc	391.2	0.2	N-Ac	15.0 (b)
Se	196.0	2.0	A-Ac	30.0 (e)
Si	251.6	0.2	N-Ac	100.0
Sm	429.7	0.2	N-Ac	300.0 (b)
Sn	286.3	0.7	N-Ac	150.0
Sr	460.7	0.4	N-Ac	5.0 (b)
Ta	271.5	0.2	N-Ac	550.0 (c)
Tb	432.6	0.2	N-Ac	300.0 (b)
Tc	261.4	0.2	A-Ac	100.0 (e)
Te	214.3	0.2	A-Ac	20.0
Ti	364.3	0.2	N-Ac	80.0
Tl	276.8	0.7	A-Ac	30.0 (e)
Tm	371.8	0.2	N-Ac	20.0 (b)
U	351.5	0.2	N-Ac	5500.0 (b)
V	318.4	0.7	N-Ac	90.0 (b)
W	255.1	0.2	N-Ac	450.0
Y	410.2	0.2	N-Ac	80.0 (b)
Yb	398.8	0.2	N-Ac	5.0 (b)
Zn	213.9	0.7	A-Ac	1.0 (e)
Zr	360.1	0.2	N-Ac	300.0 (c)

A-Ac = Air-Acetylene
N-Ac = Nitrous Oxide-Acetylene

(a) Metal concentration (mg/L) in aqueous solution which will give a reading of approximately 0.2 absorbance units.

(b) Addition of an alkali salt (e.g., K, La, or Cs as chloride) recommended to control ionization.

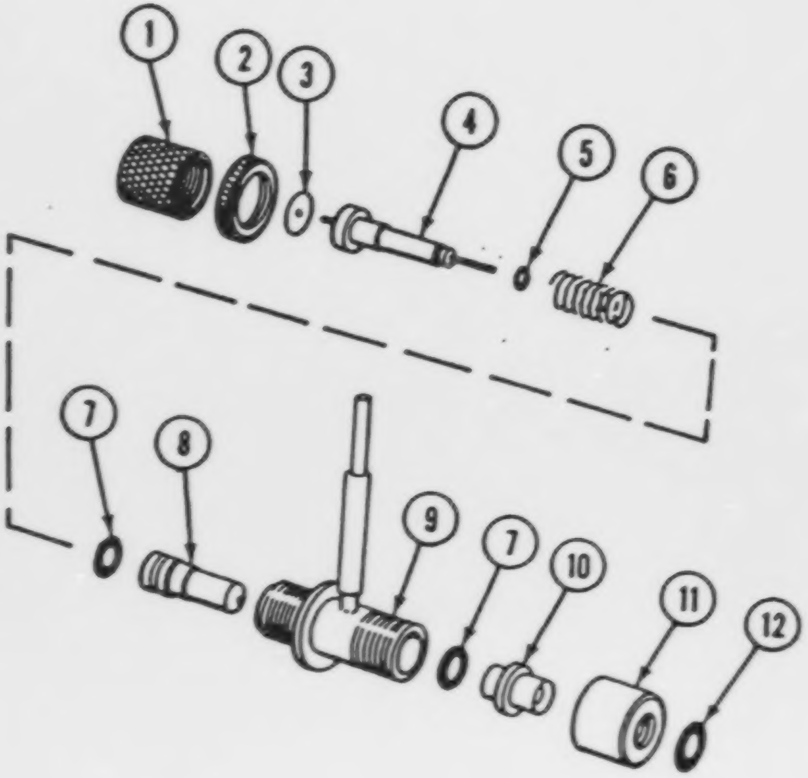
(c) 0.2% Al and 1.0% HF added to improve linearity and sensitivity.

(d) 0.1% La added to improve sensitivity.

(e) The use of the impact bead will improve sensitivity by about 2x.

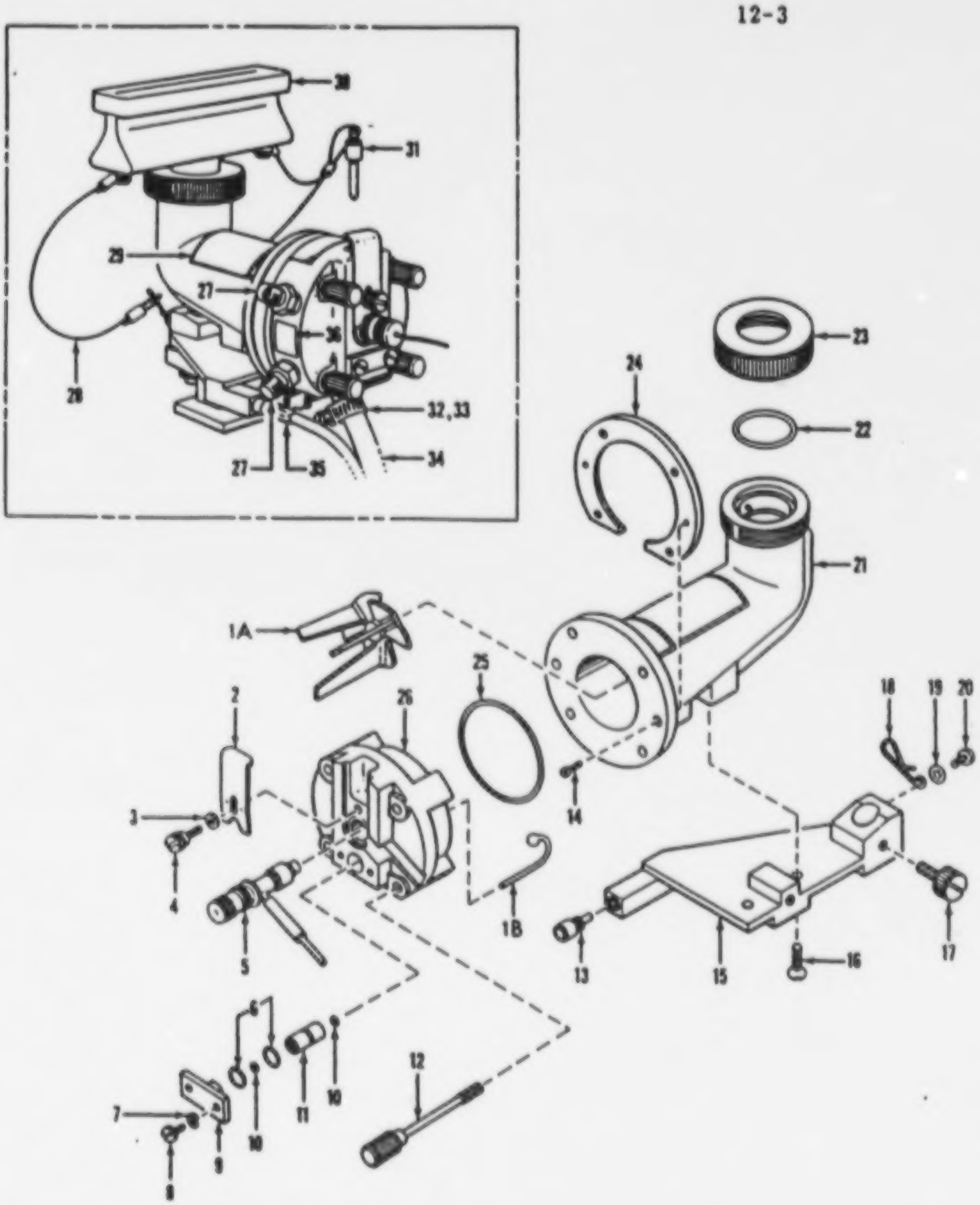
January 1982

PERKIN-ELMER



1	Knurled Knob 0303-1460*
2	Locking Ring 0303-1456*
3	Washer 0303-1802
4	Needle Assembly 0303-0354
5	O-Ring 0990-2102
6	Spring 0303-1806
7	O-Ring (2) 0990-2239
8	Insert 0303-1812
9	Nebulizer Body 0303-0353
10	Venturi 0303-1810
11	Front End Cap 0303-1808
12	O-Ring 0990-2239

Fig. 1 *Nebulizer - Exploded View*



1a) Plastic Flow Spoiler 0057-2561*
1b) Impact Bead 0057-2615*
2) Nebulizer Clamp 0057-2664***
3) Clamp Washer 0090-7731***
4) Clamp Screw 0040-1282***
5) Nebulizer Assembly 0303-0358*
6) O-Ring 0090-2240***
7) #6 Lockwasher, Split 0091-1286***
8) Screw, Binding Hd. #6-32 x 3/8 in. 0091-0148***
9) Retainer, Bead 0057-2694***
10) O-Ring 0090-2236***
11) Seal Plug, Impact Bead 0057-2624***
12) Screw, End Cap 0057-2621***
13) Interlock Connector 0057-0955*
14) Screw, Socket Hd. #4-40 x 3/8 in. 0091-0473
15) Mount Assembly 0057-0961*
16) Screw, Flat Hd #10-32 x 7/16 in. 0091-0389
17) Screw, Burner Mounting 0040-1329
18) Clip, Cable Retaining 0303-1636*
19) Washer, Flat #6 0091-1244*
20) Screw, Binding Hd. #6-32 x 3/16 in. 0091-0145*
21) Molded Burner chamber 0057-0948
22) O-Ring 0090-2038
23) Burner Head Lock Nut 0057-2696
24) Clamping Ring 0057-2616
25) O-Ring 0090-2147** ***
26) Burner End Cap 0057-2653***
27) Gas Fitting 0090-3401***
28) Retainer Cable Assembly 0303-0130*
29) Caution Label 0040-1303
30) 4-in. (10-cm) Burner Hd. 0040-0266*
31) Cable and Pin Assy, Air 0040-0376*
32) Waste Fitting 0057-2673***
33) Tubing Clamp 0090-3002*
34) Drain Tube Assembly 0047-0381*
35) Drain Interlock Connector*
36) Label, Fuel/Oxidant***

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*Not part of Burner Assembly 0057-0988

**When aspirating organic solvents, replace with Corkprene gasket 0047-2014.

***Part of End Cap Assembly 0057-0984

Figure 2. Burner Assembly (0057-0988) and Associated Parts

TABLE 2 Standard Solutions Prepara ATOMIC ABSORPTION STANDARDS

LEMENT	IRON (Fe)				MANGANESE (Mn)				SILICA (Si)				BARIUM (Ba)				STRONTIUM (Sr)				CALCIUM (Ca)			
	S1	S2	S3	REF	S1	S2	S3	REF	S1	S2	S3	REF	S1	S2	S3	REF	S1	S2	S3	REF	S1	S2	S3	REF
NDARDS (mg/L)	5.00	16.00	30.00	1.00	2.00	6.00	10.00	2.50	50.0	100.0	150.0	20.0	20.00	60.00	120.00	5.00	5.00	16.00	30.00		4.00	16.00	30.00	10.00
l of Stock	2.50	8.00	15.00	0.50	1.00	3.00	5.00	1.25	25.0	50.0	75.0	10.00	10.00	30.00	60.00	2.50	2.50	8.00	15.00		2.00	8.00	15.00	5.00
	*							*		*			*				*							*
EMICAL ADDITIVE #1	N	O	N	E	N	O	N	E	N	O	N	E	NaCl				NaCl				N	O	N	E
tock Strength													20%				20%							
l of Additive													5.0				5.0							
EMICAL ADDITIVE #2	N	O	N	E	N	O	N	E	N	O	N	E	N	O	N	E	N	O	N	E	N	O	N	E
Stock Strength																								
l of Additive																								
DILUTE TO 500 MILLILITRES WITH DEIONIZED WATER																								
EMICAL ADDITIVE #1	N	O	N	E	N	O	N	E	N	O	N	E	N	O	N	E	N	O	N	E	LaCl$_3$			
Stock Strength																					20%			
l of Additive																					5.0			
EMICAL ADDITIVE #2	N	O	N	E	N	O	N	E	N	O	N	E	N	O	N	E	N	O	N	E	N	O	N	E
Stock Strength																								
l of Additive																								
ADD 5 MILLILITRES OF CONCENTRATED NITRIC ACID																								
MARKS									Do not acidize standards or samples SiO$_2$ pp't'd in acid solutions.				NaCl need not be added to samples.				NaCl need not be added to samples				LaCl$_3$ should be added to all samples before analysing for Ca.			

sed for Sensitivity Check

TABLE 2 Standard Solutions Preparation

LEMENT	MAGNESIUM (Mg)																							
	S1	S2	S3	REF	S1	S2	S3	REF	S1	S2	S3	REF	S1	S2	S3	REF	S1	S2	S3	REF	S1	S2	S3	REF
ANDARDS (mg/L)	0.50	2.00	3.00	0.30																				
ml of Stock	0.250	1.00	1.50	0.150																				
				*																				
EMICAL ADDITIVE #1	N	O	N	E																				
Stock Strength																								
ml of Additive																								
EMICAL ADDITIVE #2	N	O	N	E																				
Stock Strength																								
ml of Additive																								
DILUTE TO 500 MILLILITRES WITH DEIONIZED WATER																								
HEMICAL ADDITIVE #1	$LaCl_3$																							
Stock Strength	20%																							
ml of Additive	5.0																							
HEMICAL ADDITIVE #2	N	O	N	E																				
Stock Strength																								
ml of Additive																								
ADD 5 MILLILITRES OF CONCENTRATED NITRIC ACID																								
EMARKS	$LaCl_3$ should be added to all samples before analysing for Mg.																							

*Used for Sensitivity Check.

METHOD W16

METALS BY EMISSION

A) **REFERENCES:** 1) Analytical Methods for Atomic Absorption Spectrophotometry, Perkin Elmer

B) **SCOPE:** This method is applicable to the analysis of surface, waste, and produced waters and any materials which can be dissolved or converted by chemical processes to aqueous solutions where the metal contents are in the mg/L range.

C) **SUMMARY:** An aqueous solution of a metal is aspirated into a burner fired by an air/acetylene mixture. The metal atoms are promoted to excited states and on returning to their ground state emit characteristic spectral bands of light. The amount of light emitted is detected and compared to a set of standards to determine the metal content of the sample.

D) **INTERFERENCES:**

1) Oil or sediment can block and foul aspirator tubes and nozzles. This is avoided by filtering the samples.
2) Flame absorbances and emissions from other elements may interfere with the emission bands of some elements. Using an alternate wave length will avoid this situation.
3) Self-absorption causes non linearity and usually occurs at high concentrations. Dilution of these strong solutions usually corrects this problem.
4) Matrix interferences result from the physical characteristics of the solvent (viscosity, high surface tension, combustible solvent). To overcome these problems the operator should use the same solvent in standards and samples, dilute high TDS samples or use the method of standard addition where all else fails. (Note #1)

E) SAFETY:

1) Acetylene:
   a) Cylinders transported on their side should be stored upright for at least 24 hours before using. Failure to do so may cause acetone/acetylene to be drawn into the regulator and result in a violent explosion.
   b) The main cylinder valve should never be opened more than 1/3 to 1/2 turn to prevent acetone escaping from the cylinder during a catastrophic line failure. It also permits quicker shutdowns under these circumstances.
   c) Acetylene cylinders should never be drawn below 520 kpa (75 psi).
   d) The acetylene delivery pressure to the A.A. should never exceed 70 kpa (10 psi) as pure acetylene spontaneously dissociates at high pressure.
   e) For more information, see Appendix A.
2) When handling hot burner heads, insulated gloves should be used to prevent burns.
3) Before using the A.A., ensure the trap on the drain line is filled with water to prevent escape of gases through the drain container.
4) Ventilation fan must be used to remove toxic combustion gases or vented fuel gases.
5) Refer to the Material Safety Data Sheets for detailed information on reagents and fuel gases.

F) APPARATUS:

1) 3 - 10 ml dispensettes with 1 litre reservoir bottles.
2) 100 ml volumetric flasks (as required).
3) 500 ml volumetric flasks (as required).
4) 500 ml plastic bottles with caps (as required).
5) Selection of pipettes suitable for making accurate dilution.
6) Perkin-Elmer 3030 Atomic Absorption Spectrophotometer.

G) REAGENTS:

1) Concentrated nitric acid
2) Acetylene
3) 1000 mg/L standard solutions

H) PROCEDURE:

a) CALIBRATION:

1) Prepare 3 calibration standards and 1 reference standard as per Table #2.
2) Turn on ventilation fan and instrument power.
3) Install the air/acetylene burner head.

4) Open the main acetylene valve 1/3 to 1/2 turn.
5) Open the air valve.
6) Set the fuel flowrate at 20 units and oxidizer flowarate at 45 units.
7) Recall the appropriate program from the User Index. (Note #2)
8) Set the slit-width and wavelength as per the method on the monitor.
9) Light the burner and allow a minimum of 15 minutes for warm-up.
10) Set the instrument on Set-up Mode and aspirate the most concentrated standard.
11) Optimize the wavelength setting, the burner vertical, horizontal and rotational settings, and the aspirator adjustment, resetting the gain as required. (See Operator's Manual).
12) Switch to an operating mode, aspirate a blank and set zero.
13) Starting with the lowest concentration, aspirate each of the calibration standards and press its calibration key. Re-zero between each standard.
14) Check the calibration by reading the reference standard. This should agree to within 5.0% of its known concentration.

b) **ANALYSIS:**

1) All samples should be preserved by acidifying with 1.0% by volume of concentrated nitric acid.
2) Re-zero the instrument using a blank.
3) Add any acids and reagents as listed in the Standard Conditions (Table 1) for the element in question.
4) Samples with oil or suspended solids require filtration before analysis.
5) Aspirate the sample and read the concentration from the monitor.
6) If the sample is too concentrated, make an accurate dilution using pipettes and volumetrics, re-treat with the appropriate reagents and repeat the analysis.
7) After every 5 samples, run a calibration standard to ensure accuracy of calibration.

I) **CLEANING:**

1) Glassware:
   a) Normal laboratory procedure should be adequate.

2) Aspirator (Minor Cleaning):
   a) If the aspirator becomes clogged with oil or sediments it can frequently be cleared by passing a wire through the aspirator tubing and assembly. (Note #3)
   b) Re-check the calibration before continuing with analysis.

3) Aspirator (Major Cleaning):
   a) If the above fails to clear the problem, shut down the burner and dismantle the aspirator assembly.
   b) Wash the metal parts and tubing with solvent and/or soap and water.
   c) Use the cleaning wire to clear any debris from the tubing or needle.
   d) Re-assemble the aspirator (Figure #1).
   e) Re-light and re-warm the burner, adjust the aspirator and recalibrate before continuing the analysis.

4) Burner Heads:
   a) All burner cleaning requires shutting down of the burner. (Note #4).
   b) Carbon, salt and other deposits on the burner slot can be removed using the razor blade or scraper tool provided.
   c) More tenacious deposits can be removed by soaking the head in water and brushing with a bottle brush and using the scrapers.
   d) Burner heads are to be cleaned at the completion of each sample batch.

5) Premix Chamber:
   a) After completion of each batch of samples the premix chamber should be flushed with distilled water to remove salt solutions and deposits.
   b) If the walls are oily, the burner assembly should be dismantled, solvent washed, wiped clean and water rinsed. The unit is then re-assembled. (Figure #2)

J) CALCULATIONS:

1) $C = \frac{V \times A}{S}$

V = Final dilution volume, ml
S = Sample aliquot, ml
A = Instrument reading, mg/L
C = Original sample concentration, mg/L

K) PRECISION:

1) Duplicate results shall agree to within 5.0 % of each other.

L) QUALITY CONTROL:

1) Results which fail the precision criterion shall be repeated.
2) A calibration check shall be run after every fifth sample, utilizing the standard closest to the last sample analyzed.
3) Failure of the standard to fall within 5.0 % of

its known value requires the operator to recalibrate and repeat the five previous analysis with another calibration check included.

M) REPORTING:

a) Significant Figures: (See Technical Section)

1) All results shall be reported to 3 significant figures.

b) Format: 1) The following format is recommended:

Metal, mg/L	XX.X
eg. Calcium, mg/L	1 230.

N) NOTES:

1) See the Operator's Manual for the method of standard additions. This approach is rarely used at this time and therefore does not warrant being published and is only included for completeness.
2) See the Operator's Manual for selecting and initializing new user index programs.
3) This can be done while the instrument is operating, avoiding a shut down and re-warming of the burner.
4) Exercise care in handling hot burners. Use insulated gloves and avoid contact with hot components in the burner compartment.

REVISION DATE: 1989-10-30

METHOD W17

CONDUCTIVITY (Radiometer)

(CDM83 Conductivity Meter)

A) **REFERENCES:**

1) Operating Instructions Manual CDM 83 Conductivity Meter.

B) **SCOPE:**

This method is used to detect the conductivity of all types of water samples.

C) **SUMMARY:**

The water sample is brought to the temperature of interest and the conductivity of the water sample is read by inserting the conductivity cell into the water.

D) **INTERFERENCES:**

1) Oil in the sample fouls the cell and results in inaccurate readings. Filter the sample to remove the oil from the water.

E) **SAFETY:**

1) No hazards worth noting.

F) **APPARATUS:**

1) CDM 83 Conductivity Meter
2) T801 Temperature Sensor
3) Conductivity Cell (CDC304 Immersion)
4) Stand to hold conductivity cell and temperature sensor (E186 Electrode Stand).
5) 100 ml Graduated Cylinder or other suitable container for the water to be tested.
6) Beaker for storing conductivity cell and temperature sensor. (Note #1)

G) **REAGENTS:**

1) Distilled Water
2) 0.05% Sodium Chloride (NaCl) - Weigh 0.5000 g NaCl into a 1 liter volumetric flask, dissolve and dilute to 1 liter with deionized water.

H) PROCEDURE:

a) CALIBRATION:

1) Immerse the conductivity and temperature sensor into a sample of 0.05% NaCl that has been brought to the temperature of interest.
2) Press AUTORANGE.
3) Press the MAN and MAN keys simultaneously.
4) The display on the CDM83 now shows "CALIBRATED" for 2 seconds. Thereafter, the CDM83 will display the actual conductivity of the 0.05% NaCl and the calculated cell constant simultaneously. (Note #2, 3, and 4)

b) ANALYSIS:

1) The water sample to be tested is filtered to remove oil and all foreign material.
2) The filtered sample is brought to the temperature of interest (usually 25°C).
3) Immerse the conductivity cell and the temperature sensor into the water sample.
4) Press AUTORANGE.
5) Record the value displayed on the CDM83 in ms/cm and the temperature when display is stable.

I) CLEANING:

1) The conductivity cell and temperature sensor shall be rinsed with distilled water between samples.

Occasionally it may be necessary to clean the conductivity cell and temperature sensor more thoroughly.

J) CALCULATIONS:

1) 1 mS/cm = 1000 μ/cm
Therefore, multiply all readings in mS/cm x 1000

K) PRECISION:

1) Accurate to ±0.75% ±3 in least significant digit in the most common ranges (13 μS/cm - 130 mS/cm).

L) QUALITY CONTROL:

1) Check the cell constant and calibration each time the equipment is used or weekly, whichever is most applicable.

Duplicate samples should agree within precision requirements. Repeat if necessary.

M) REPORTING:

a) Significant Figures:

1) All results shall be reported to 3 significant figures.

b) Format: 1) Report As:

Conductivity, μS/cm @ XX.X°C XXX

N) NOTES:

1) Cell must not be allowed to dry out. Store cell in distilled water.
2) The temperature coefficient (T.C.%) has been automatically reset to zero.
3) This procedure also is used to check the cell constant.
4) Conductivity of 0.05% NaCl at 25°C in μS/cm is 1015.

REVISION DATE:

METHOD W18

RESISTIVITY

A) **REFERENCES:** 1) Instructions for USI Model 31 Conductivity Bridge.

B) **SCOPE:** This method is used to read the resistance of all types of water samples.

C) **SUMMARY:** The water sample is filtered to remove oil and brought to the temperature of interest. The conductivity/resistivity is immersed in the water and resistance is measured.

D) **INTERFERENCES:** 1) Oil interferes with the measurement by coating the probe and making it difficult to make an accurate reading. Filter the sample before testing if necessary.

E) **SAFETY:** 1) No known hazards.

F) **APPARATUS:**

1) Model 31 Conductivity Bridge
2) Conductivity Cell (#3401 dipping type)
3) Beaker
4) 100 ml Graduated Cylinder or other suitable container

G) **REAGENTS:**

1) Distilled Water
2) Calibration Standard - Dissolve 0.745 grams of pure dry potassium chloride (KCl) in distilled water and dilute until the total solution weighs 1 kg.

H) PROCEDURE:

a) CALIBRATION:

1) Set the Function switch to LINE and allow to warm up for at least 5 minutes.
2) Measure the conductivity of distilled water and the calibration standard at various temperatures. To do this:
    a) Immerse the cell in the distilled water at the temperature in question.
    b) Set the Sensitivity control to minimum.
    c) Set the Range switch to the conductance range which gives the maximum shadow width on the indicator tube. (Note #1)
    d) Adjust the Drive control for the widest shadow.
    e) If the dial indication is above 10.0 or below 1.0, set the Range switch to the next higher or lower range, whichever is appropriate, and readjust the Drive control for the widest shadow.
    f) Set the Sensitivity control to maximum and readjust the Drive control for maximum shadow.
    g) The conductivity reading is the conductance reading times the multiplier which is in line with the pointer.
    h) Switch the Range switch to read the resistance of the sample.
    i) Record all readings.
    j) Calculate the cell constant.

b) ANALYSIS:

1) Bring sample to test temperature.
2) Immerse the conductivity cell in the sample.
3) Set Range switch to read the resistance of the sample.
4) Set the Sensitivity control to the minimum.
5) Set the Range switch to the resistance range which gives the maximum shadow width on the indicator tube.
6) Adjust the Drive control for the widest shadow.
7) If the dial indication is abo e 10.0 or below 1.0, set the Range switch to the next higher or lower range, whichever is appropriate, and re-adjust the Drive control for the widest shadow.
8) Set the Sensitivity control to maximum and re-adjust the Drive control for maximum shadow.
9) The resistivity reading is the resistance reading times the multiplier which is in line with the pointer.

10) Record the reading. (Note #4)

I) CLEANING, STORAGE & MAINTENANCE OF CONDUCTIVITY CELL:

1) Between samples rinse the conductivity cell with distilled water.
2) Whenever necessary, clean the cell with a solution of the following to remove contamination:
   a) Prepare a solution of 100 ml isopropyl alcohol, 100 ml petroleum ether, 50 ml of concentrated HCl and 50 ml of distilled water.
   b) Immerse the cell past the vent slots in this solution.
   c) Stir the cell in this solution for 1 or 2 minutes.
   d) Remove the cell from the solution and rinse thorougly with distilled water.
   e) Repeat if necessary.
3) Store the conductivity cell in distilled water or disconnect it from the bridge. Never store a dirty cell.
4) The electrodes of the cell are coated with platinum black before calibration. A poor coating of latinum black will cause the shadow on the indicator tube to be blurred and indistinct.

J) CALCULATIONS:

1) Cell Constant

$$K = \frac{R(C_1 + C_2)}{10^6}$$

Where K = cell constant

R = measured resistance in ohms.cm

C_1 = conductivity in µS/cm of solution

C_2 = conductivity in µS/cm of distilled water used to make solution

K) PRECISION:

1) Duplicate results are accurate to ±1% in the range of 2.0 ohm-cm to 2.0 megaohm-cm. Accuracy decreases outside of this range.

L) QUALITY CONTROL:

1) Repeat samples that fall out of the precision range.

M) REPORTING:

a) Significant Figures:

1) All results shall be reported to 3 significant figures.

b) Format: 1) Report As:

Resistivity, ohm-cm @ XX.X°C XXX

N) NOTES:

1) If a wide, well-defined shadow indication cannot be obtained, set the Function switch to the 1 KHz position.
2) Conductivity bridge reads conductance in micromhos/cm. In metric terms micromhos/cm are equal to microSiemens/centimeter (μS/cm).
3) Conductivity bridge reads resistance in ohms. In metric terms ohms are equal to ohm-cm.
4) The conductivity and resistivity readings can be compared by the following relationships:

$$\text{Conductivity} = \frac{1}{\text{RESISTIVITY}} \times 1{,}000{,}000$$

REVISION DATE:

APPENDIX 2. THE DEAN STARK TECHNIQUE

SYNCRUDE
ANALYTICAL METHODS FOR OIL SAND AND BITUMEN PROCESSING

SYNCRUDE CANADA LIMITED
EDMONTON, ALBERTA, CANADA
August 1979

Determination of Bitumen, Water and Solids Content of Middlings and Tailings Samples (Classical)

Scope: This method determines the bitumen, water and solids content of middlings and tailings samples. Preferred sample sizes range from 250 to 600 g, although the method may be modified for smaller samples.

Summary: The sample is separated into bitumen, water and solids by refluxing with toluene in a solids extraction apparatus. Condensed solvent and co-distilled water are continuously separated in a trap. The water is drained from the lower portion of the trap while the solvent recycles through the extraction thimble to dissolve bitumen. When the extraction is complete, the clean solids retained in the thimble are dried and weighed. A density measurement is employed to determine the bitumen content of the extract. Water is determined by difference.

Safety Considerations:

1. Hydrochloric acid — highly toxic by inhalation. A strong irritant to eyes and skin.
2. Toluene — moderately toxic by inhalation and skin absorption. Possesses irritant and anesthetic properties. Highly flammable.

Apparatus, Reagents, Materials:

1. Condenser — an indented tube condenser having a water jacket at least 535 mm long with a 29/42 standard taper male joint at the base.
2. Water trap — constructed to continuously separate water and solvent. The trap portion, which is approximately 25 mm in diameter and 220 mm long, has a volume of about 130 mL. The lower end is fitted with a stopcock for removal of water while the upper section has a 29/42 standard taper female joint to accommodate the condenser. The downcomer has a 103/60 standard taper male joint for connection to the kettle and is perforated with 3 mm diameter holes below the ground glass.
3. Kettle — a heat-resistant glass vessel having a capacity of 2000 mL. The neck has a minimum length of 390 mm and includes a 103/60 standard taper female joint.
4. Extraction thimble — Whatman 85 x 200 mm cellulose thimble, single thickness.
5. Thimble basket — a corrosion-resistant basket to support the thimble. See Figure 1. Fabrication from 3 mm diameter nichrome wire using a silver solder bottom joint is suitable.
6. Solvent distributor — corrosion-resistant, as shown in Figure 2.
7. Heating mantle — 500 W, to accommodate 2000 mL kettle.

The assembled extraction apparatus is shown schematically in Figure 3. If samples are less than 100 g, then the extraction apparatus described in Method 2.7 is used with the exception that the lower end of the water trap is fitted with a drain cock. Volumes employed in the following procedure are then gauged accordingly.

8. Kimwipes — Type 1300.
9. Supply of 800 mL beakers.
10. Volumetric flasks — 250 and 1000 mL, Class A.
11. Syringe — 5 mL, glass, Luer-Lok tip.
12. Disposable filter assemblies — Millipore Millex filters, 0.45 µm, 25 m diameter, catalogue no. SLHA 025 OS.
13. Densimeter — six place densimeter with constant temperature water bath fo temperature control to ±0.01°C. The water bath is operated at 20°C.
14. Balances:
    Top loading balance to ±0.01 g, 1200 g capacity.
    Analytical balance.

15. Drying oven — forced draft, set at 120°C.
16. Desiccator.
17. Reagents:
    Hydrochloric acid, A.C.S. reagent grade.
    Toluene, A.C.S. reagent grade.
18. Distilled water.

Procedure: Sample Handling

1. Insert 3 Kimwipes into each of a quantity of thimbles. Place the thimbles into 800 mL beakers and weigh to the nearest 0.01 g.
2. Clean the outer walls of the sample container and lid. Weigh to the nearest 0.1 g. Carefully add 0.5 mL of concentrated hydrochloric acid to the sample. Then add approximately 100 mL of toluene and stir until all the bitumen adhering to the wall of the sample container is dissolved.
3. Remove the Kimwipes from the thimble, insert the thimble into a thimble basket and mount the basket by its support into the neck of a kettle. Add approximately 900 mL of toluene to the kettle by pouring it through the thimble.
4. Restir the sample and while it is still in motion transfer to the thimble. Wash the container with toluene to remove all the bitumen. Add the washings to the thimble. Transfer the bulk of the remaining solids by scraping with a spatula. Finally, rinse and transfer remaining solids with distilled water. Clean and dry the container and lid with the Kimwipes and place them in the thimble. Weigh the container and lid.

Extraction

5. Assemble the extraction apparatus as shown in Figure 3, ensure all joints are vapour tight, and begin refluxing. Drain and discard water from the trap when it nears the trap overflow. The extraction is complete when the level of water in the trap is constant and solvent dripping from the thimble is clear. Terminate the extraction and allow the apparatus to cool.

Calibration of Densimeter

6. Prepare a series of standard solutions of middlings and tailings bitumen in toluene covering the range 0.3 - 12 g/250 mL. (a)
7. Filter the standards through a 0.45 μm filter and determine the density of the solutions to six places.
8. Prepare a calibration curve by plotting the density of the standards versus the bitumen concentration in grams of bitumen per 250 mL. Calculate the slope and intercept of the plot using a linear least square regression. Coefficients of determination better than 0.999 are expected. The calibration does not have to be repeated daily, but several points on the calibration curve should be checked periodically to confirm the calibration.

Bitumen Determination

9. Disassemble the extraction apparatus and pour the bitumen/toluene extract into a 1000 mL volumetric flask and dilute to volume with toluene. Filter several milliliters of the solution through a 0.45 μm filter and determine its density to six decimal places. Ensure that the measured density falls within the range of the calibration.

Solids Determination

10. Remove the thimble from the extraction apparatus, place it in its beaker and dry for 12 hours at 120°C. Cool and reweigh.

Calculations: The bitumen, water and solids contents of the sample are given by:

$$\% \text{ Bitumen} = \frac{B}{W} \times 100$$

$$\% \text{ Solids} = \frac{S}{W} \times 100$$

$$\% \text{ Water} = 100 - (\text{wt } \% \text{ Bitumen} + \text{wt } \% \text{ Solids})$$

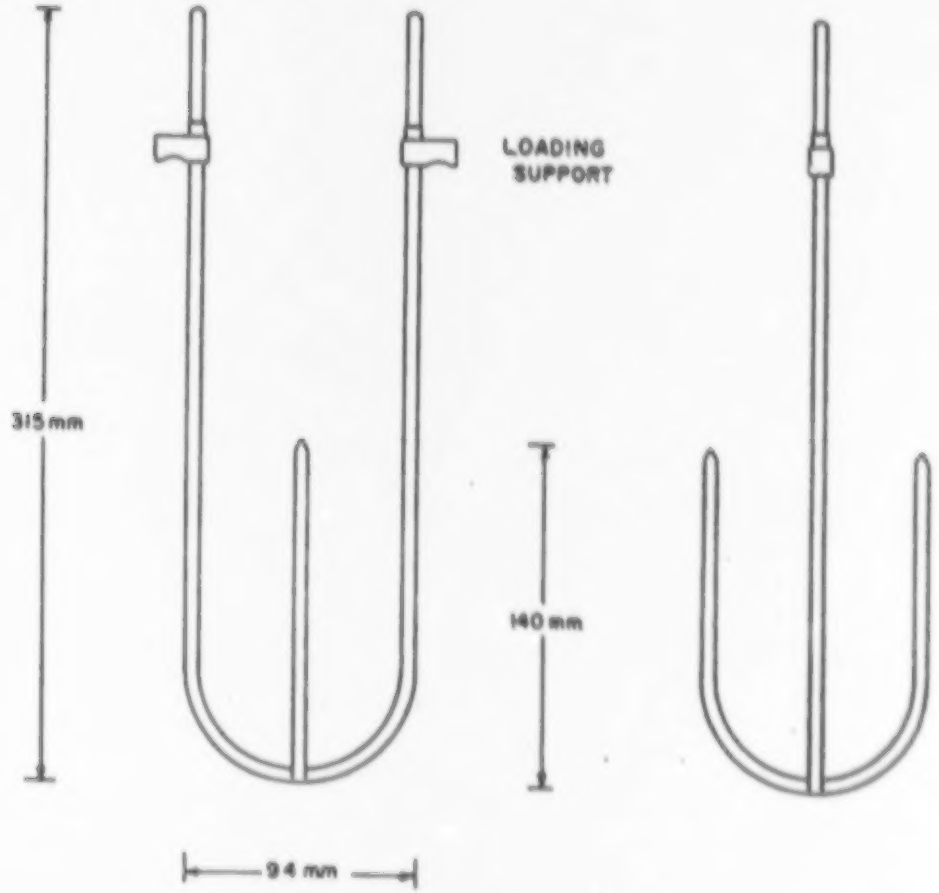
where: W = weight of sample, g
S = weight of solids recovered in thimble, g
$$B = \frac{\text{Sample Density} - \text{Calibration Intercept}}{\text{Calibration Slope}} \times 4$$
= weight of bitumen in sample, g

Precision: Data tabulated below indicates typical precision values associated with the bitumen water and solids analysis of middlings and tailings samples. Estimates reflecting th repeatability of a middlings analysis were determined on 37 replicate subsample prepared by Method 1.6 and analyzed using the small extractor assemblies. Precisio values for primary and secondary tailings were evaluated from 10 replicate sample obtained in rapid succession from pilot plant process streams and analyzed using larg extractor assemblies. All data includes errors due to sampling.

	Mean Value (wt%)	Standard Deviation (wt%)	Relative Standard Deviation
Middlings			
Bitumen	7.32	±0.15	2.05%
Water	75.91	±0.42	0.55%
Solids	16.77	±0.38	2.27%
Primary Tailings			
Bitumen	0.87	±0.06	6.90%
Water	35.01	±1.47	4.20%
Solids	64.12	±1.48	2.31%
Secondary Tailings			
Bitumen	0.44	±0.02	4.55%
Water	79.98	±1.71	2.14%
Solids	19.58	±1.71	8.73%

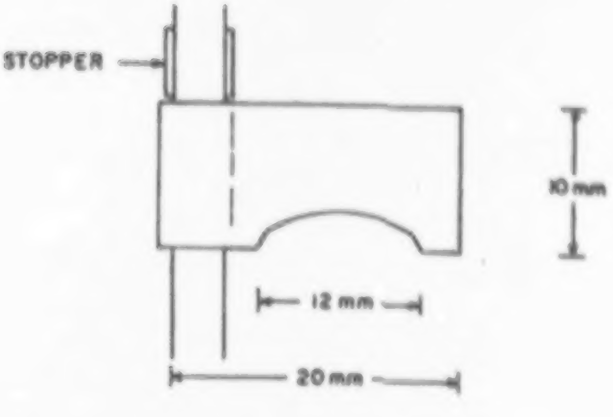
Comments:

a. The bitumen for the standard solutions is prepared by extracting a large numb of middlings and tailings samples on a solids extraction apparatus, removir non-filtered solids by centrifuging, removing toluene by evaporation, ar determining the residual toluene content by the procedure described in Metho 2.7.



FRONT VIEW

SIDE VIEW



LOADING SUPPORT

FIGURE 1: EXTRACTION THIMBLE BASKET

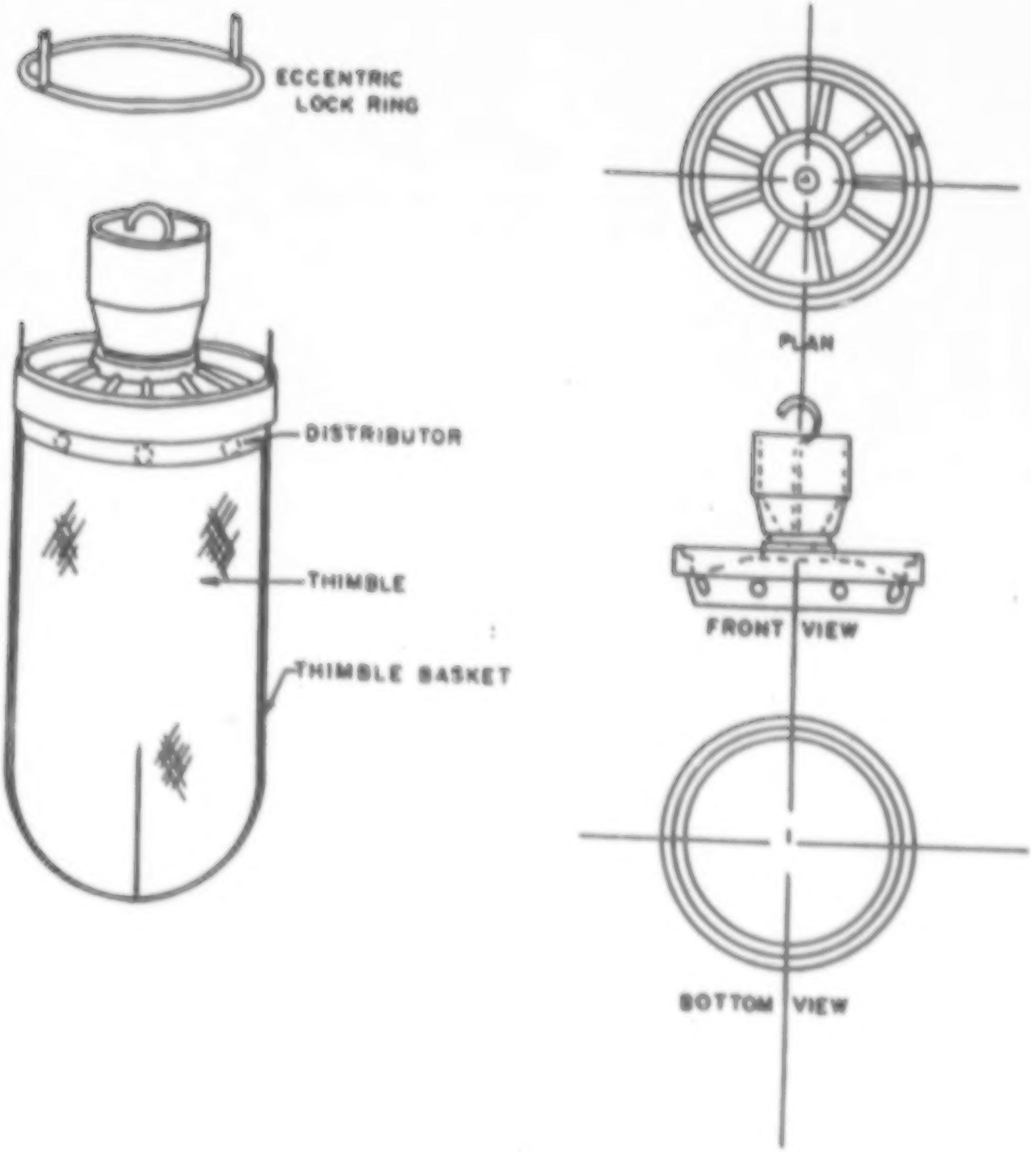


FIGURE 2: THIMBLE ASSEMBLY AND SOLVENT DISTRIBUTOR

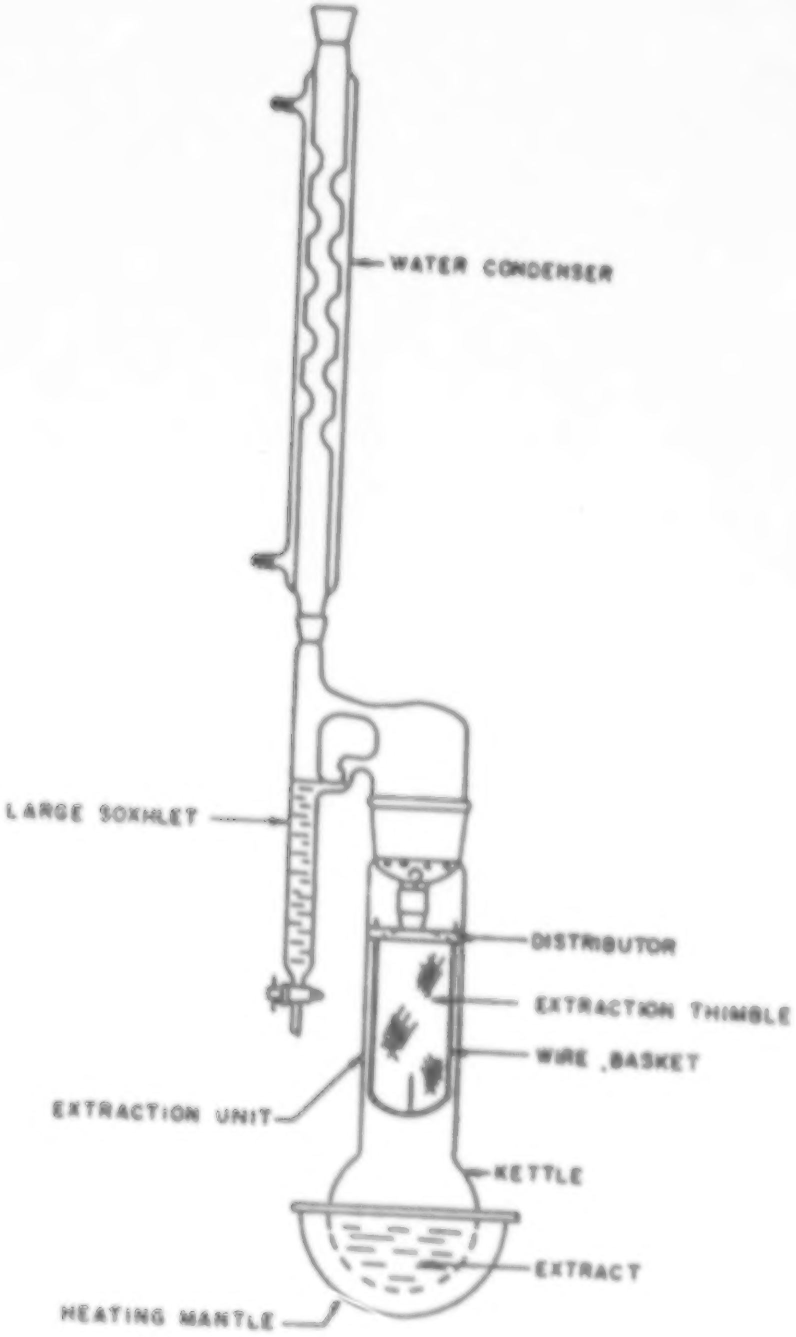


FIGURE 3: EXTRACTION APPARATUS

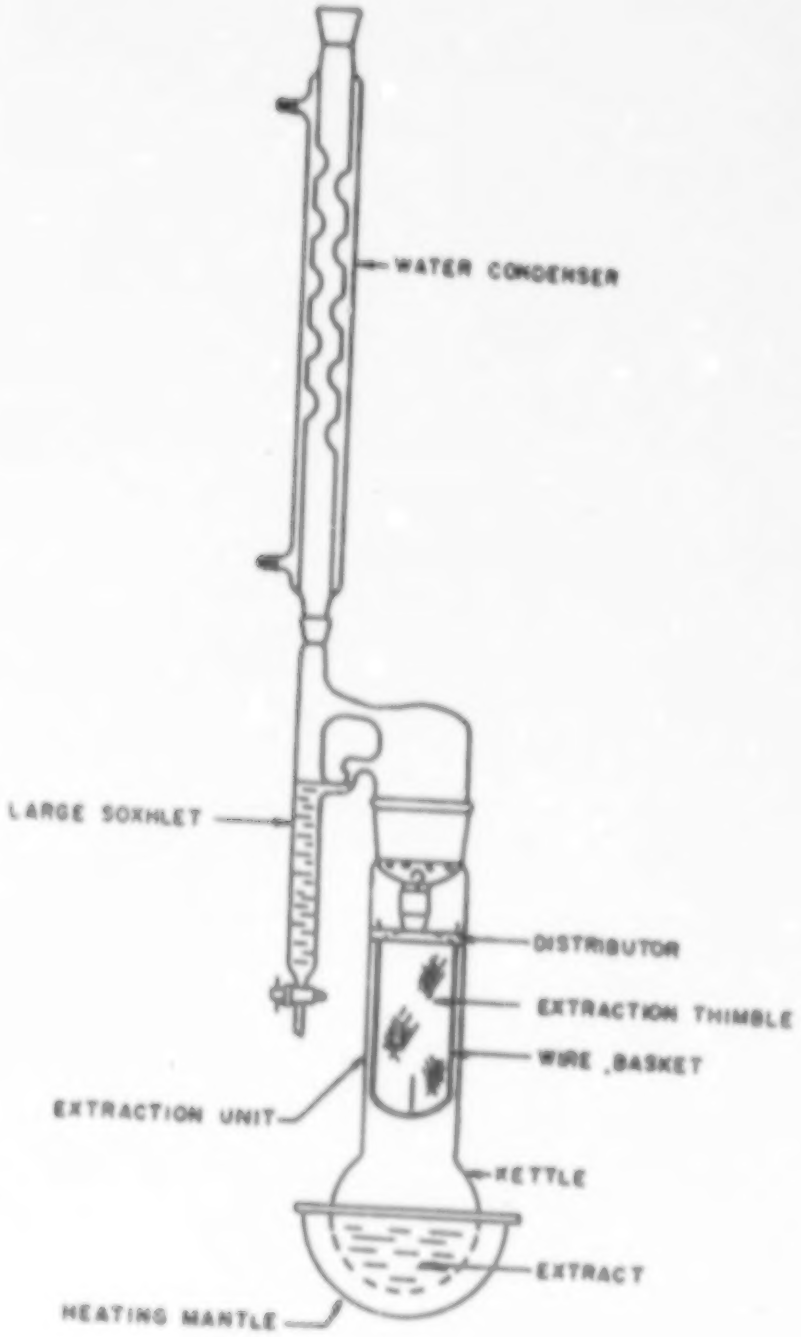


FIGURE 3: EXTRACTION APPARATUS

APPENDIX 3. WEIGHTS OF OIL RECOVERED FROM THE BIOREACTOR

DATE AND SAMPLE LOCATION	EXTRACTED WEIGHTS		
	OIL (g)	SOLID (g)	OIL/SOLID (g/g)
JAN 19/90			
1 upper	10.64	49.86	0.21
1 lower	6.58	33.80	0.19
6 upper	8.54	41.66	0.21
6 lower	9.18	41.75	0.22
2 upper	18.13	24.88	0.73
2 lower	18.03	23.88	0.76
3 upper	17.94	22.27	0.81
3 lower	18.72	25.54	0.73
4 upper	19.10	22.50	0.85
4 lower	17.07	20.16	0.85
5 upper	15.99	19.09	0.84
5 lower	14.88	23.33	0.64
MAR 19/90			
1 upper	5.75	45.45	0.13
1 lower	6.46	41.23	0.16
6 upper	6.19	45.68	0.14
6 lower	6.83	46.41	0.15
2 upper	7.58	15.36	0.49
2 lower	12.97	21.45	0.60
3 upper	9.80	18.72	0.52
3 lower	12.70	23.54	0.54
4 upper	15.11	21.24	0.71
4 lower	17.34	32.40	0.54
5 upper	13.30	21.52	0.62
5 lower	14.69	23.47	0.63
APR 2/90			
1 upper	4.35	41.27	0.11
1 lower	6.69	49.79	0.13
6 upper	6.07	46.31	0.13
6 lower	5.30	44.99	0.12
2 upper	12.83	22.57	0.57
2 lower	13.52	24.29	0.56
3 upper	17.76	23.94	0.74
3 lower	16.36	26.39	0.62
4 upper	20.57	25.92	0.79
4 lower	14.56	24.57	0.59
5 upper	14.30	25.89	0.55
5 lower	12.28	26.11	0.47

DATE AND SAMPLE LOCATION	EXTRACTED WEIGHTS		
	OIL (g)	SOLID (g)	OIL/SOLID (g/g)
APR 16/90			
1 upper	5.34	49.77	0.11
1 lower	6.85	51.32	0.13
6 upper	5.23	48.75	0.11
6 lower	5.83	50.42	0.12
2 upper	13.20	23.60	0.56
2 lower	11.35	22.69	0.50
3 upper	16.46	25.95	0.63
3 lower	16.13	25.65	0.63
4 upper	17.62	29.05	0.61
4 lower	14.37	26.18	0.55
5 upper	12.36	25.75	0.48
5 lower	10.87	23.49	0.46
MAY 28/90			
1 upper	4.18	49.07	0.09
1 lower	6.33	51.47	0.12
6 upper	4.09	49.45	0.08
6 lower	4.26	51.19	0.08
2 upper	10.55	25.55	0.41
2 lower	9.98	24.78	0.40
3 upper	12.65	26.57	0.48
3 lower	13.25	29.96	0.44
4 upper	12.92	28.06	0.46
4 lower	13.60	30.78	0.44
5 upper	11.80	28.25	0.42
5 lower	13.52	29.20	0.46
JUNE 13/9			
1 upper	5.04	52.65	0.10
1 lower	4.71	54.89	0.09
6 upper	4.02	55.23	0.07
6 lower	3.35	55.07	0.06
2 upper	10.07	25.43	0.40
2 lower	10.90	28.93	0.38
3 upper	14.78	29.68	0.50
3 lower	11.95	31.35	0.38
4 upper	11.63	26.20	0.44
4 lower	12.39	29.81	0.42
5 upper	9.15	25.73	0.36
5 lower	11.58	27.37	0.42

APPENDIX 4. GAS CHROMATOGRAMS OF THE OIL RECOVERED FROM THE BIOREACTOR

FIGURE 1
GC-FID CHROMATOGRAM OF #1 UPPER BIOREACTOR
MAY 28, 1990

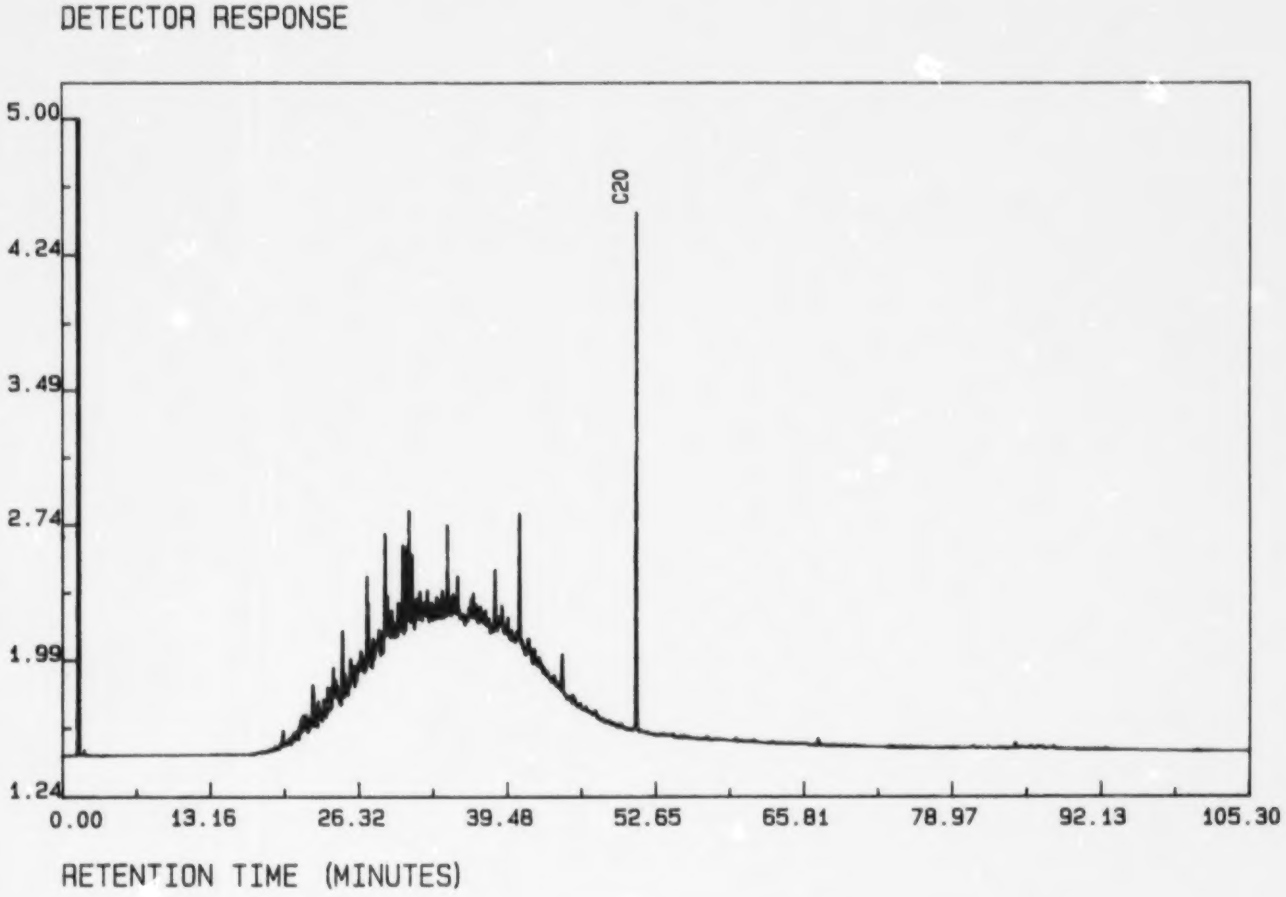


FIGURE 2

GC-FID CHROMATOGRAM OF #6 UPPER BIOREACTOR

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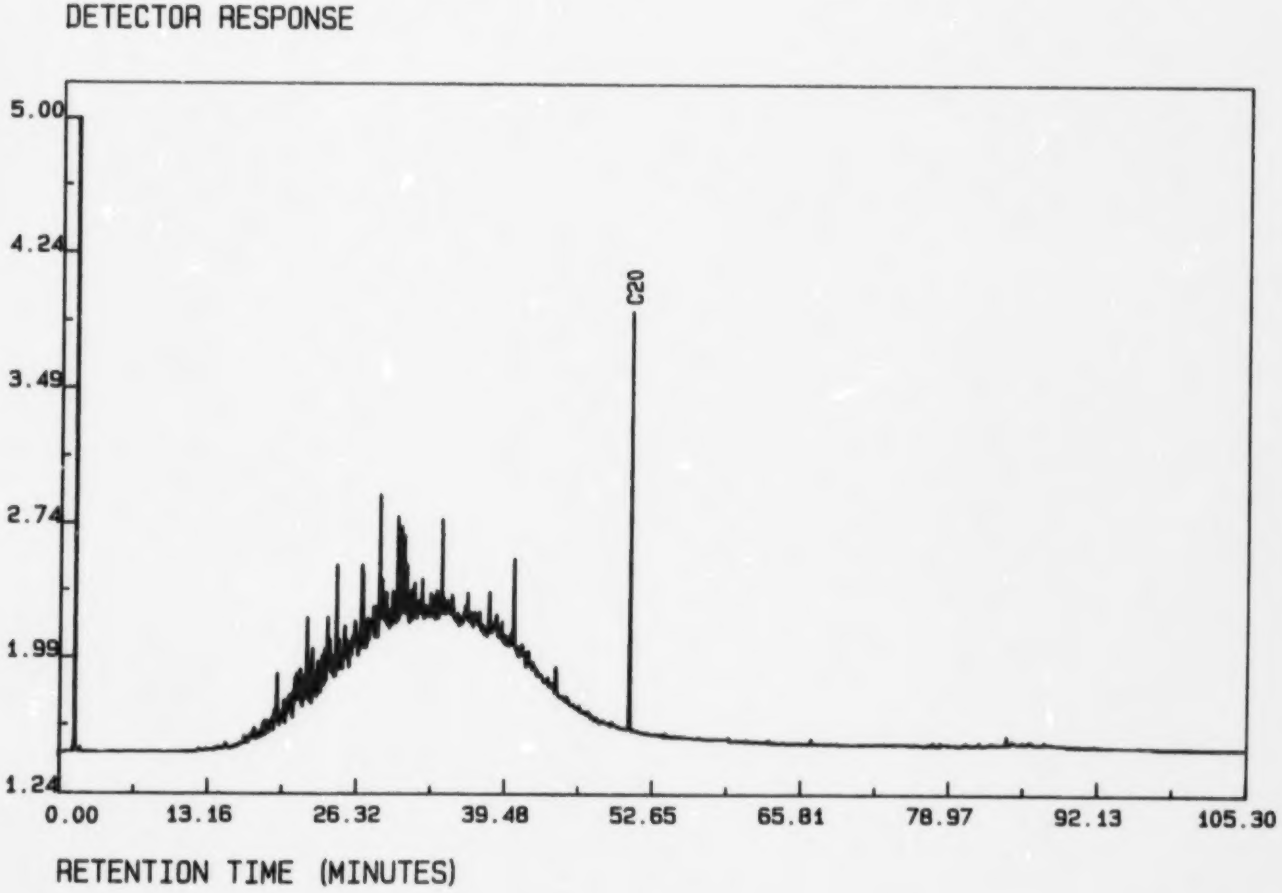

FIGURE 3
GC-FID CHROMATOGRAM OF #1 LOWER BIOREACTOR
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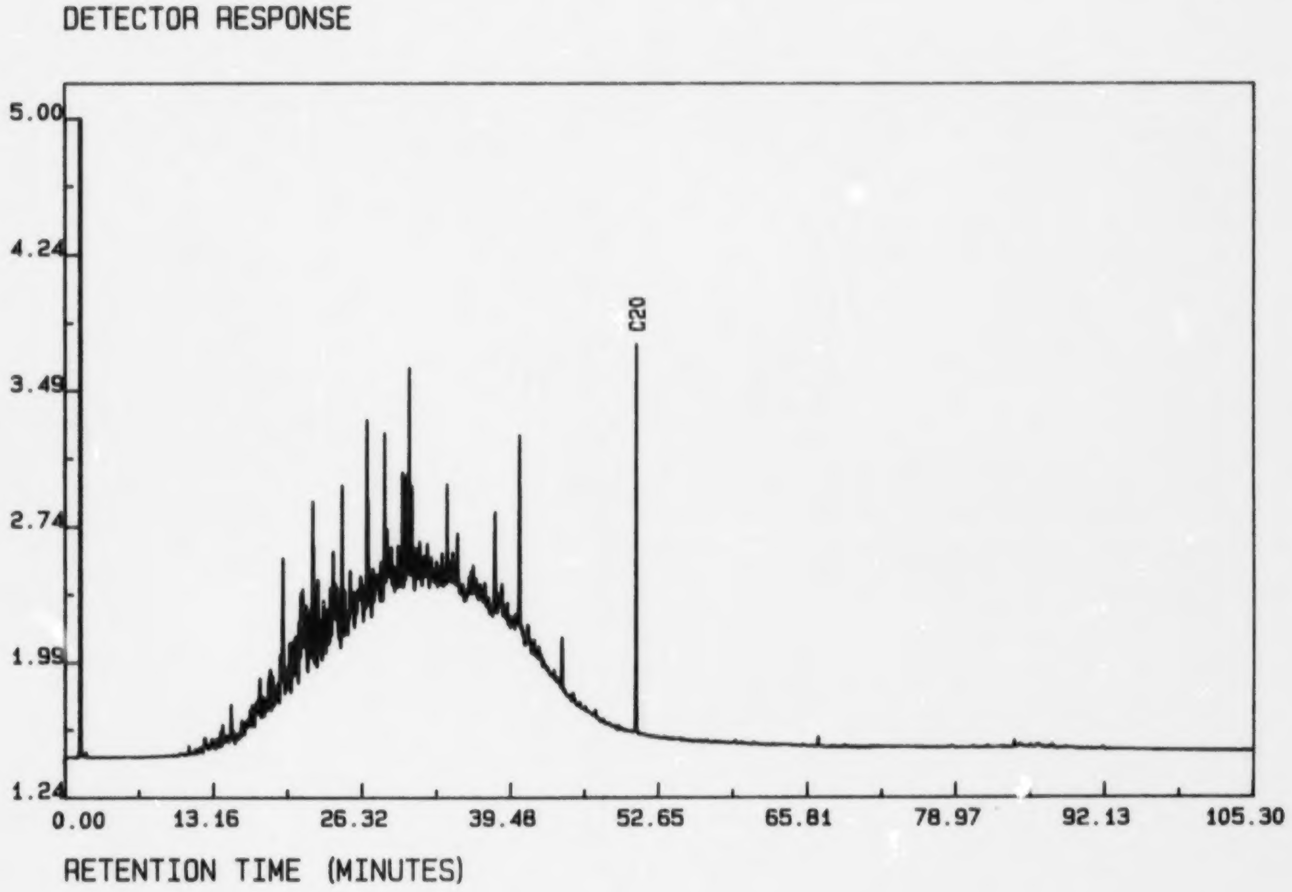

FIGURE 4

GC-FID CHROMATOGRAM OF #6 LOWER BIOREACTOR

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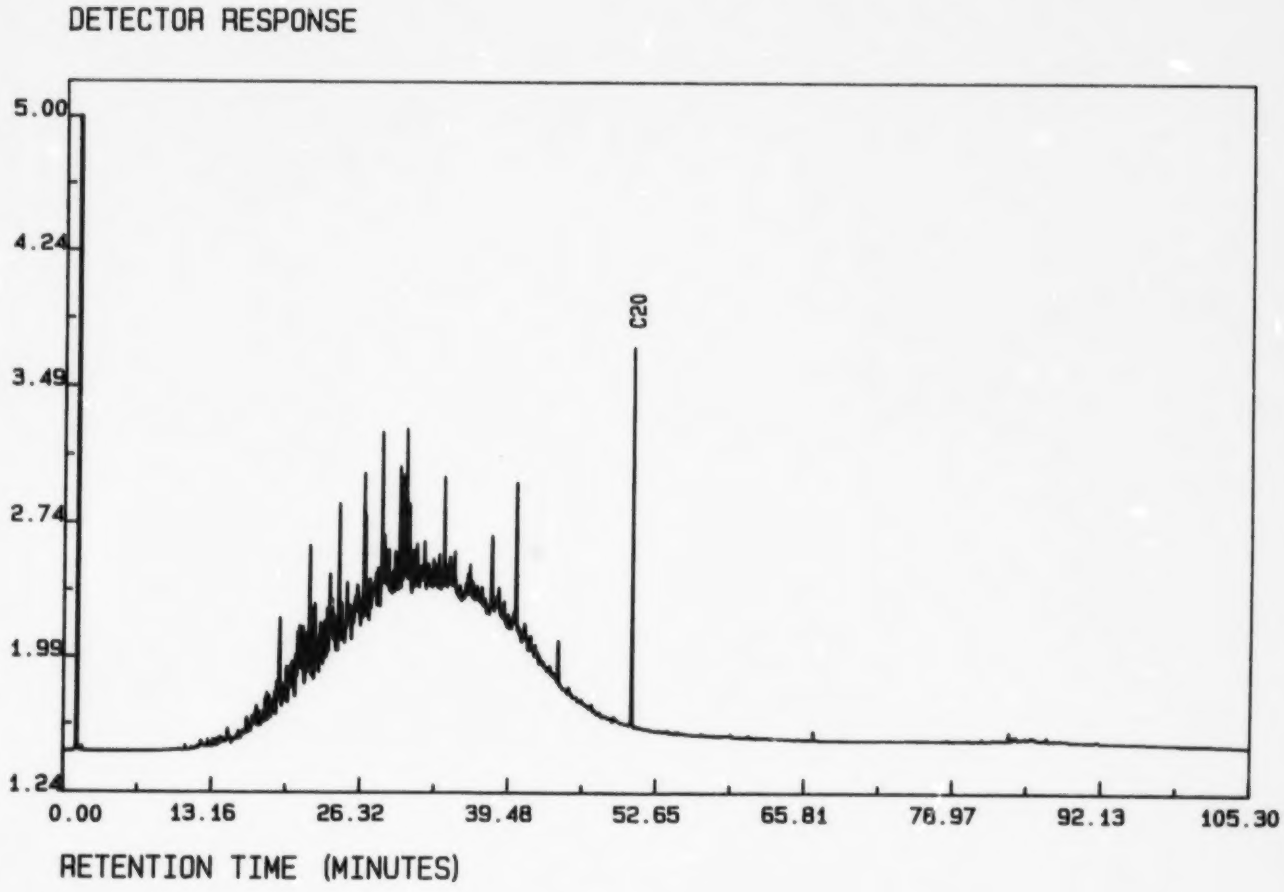

FIGURE 5

GC-FID CHROMATOGRAM OF #1 UPPER BIOREACTOR

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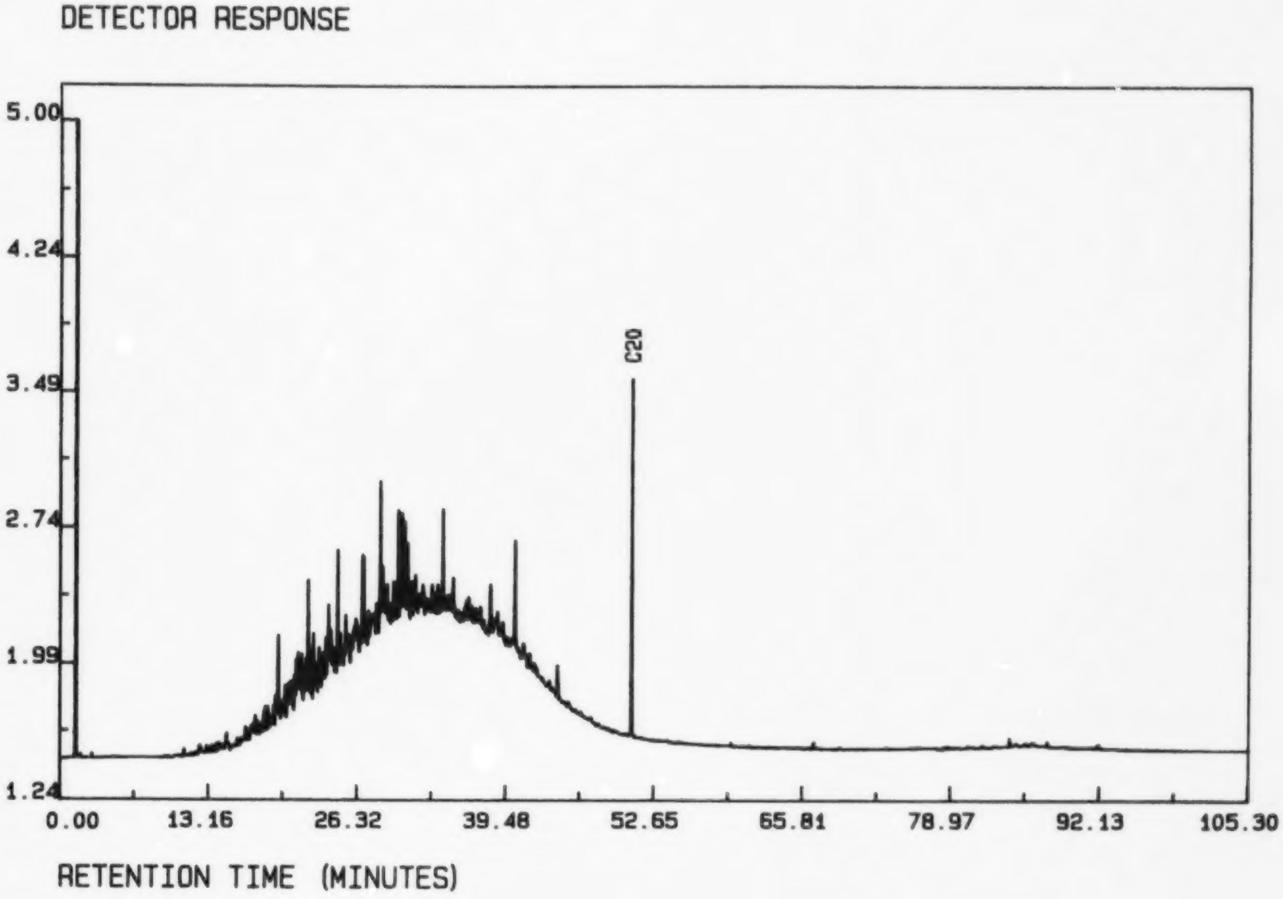

FIGURE 6

GC-FID CHROMATOGRAM OF #6 UPPER BIOREACTOR

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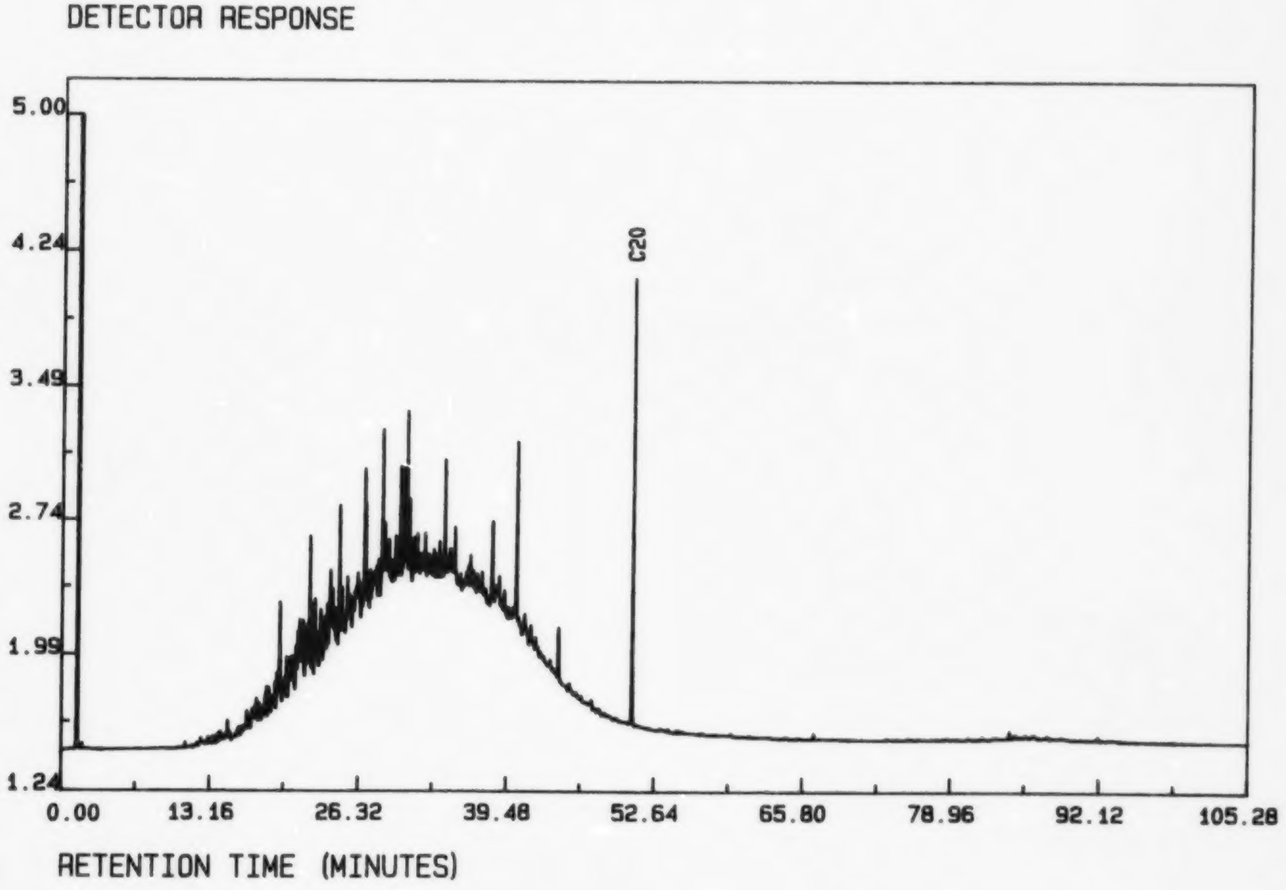

FIGURE 7

GC-FID CHROMATOGRAM OF #1 LOWER BIOREACTOR

JUNE 13, 1990

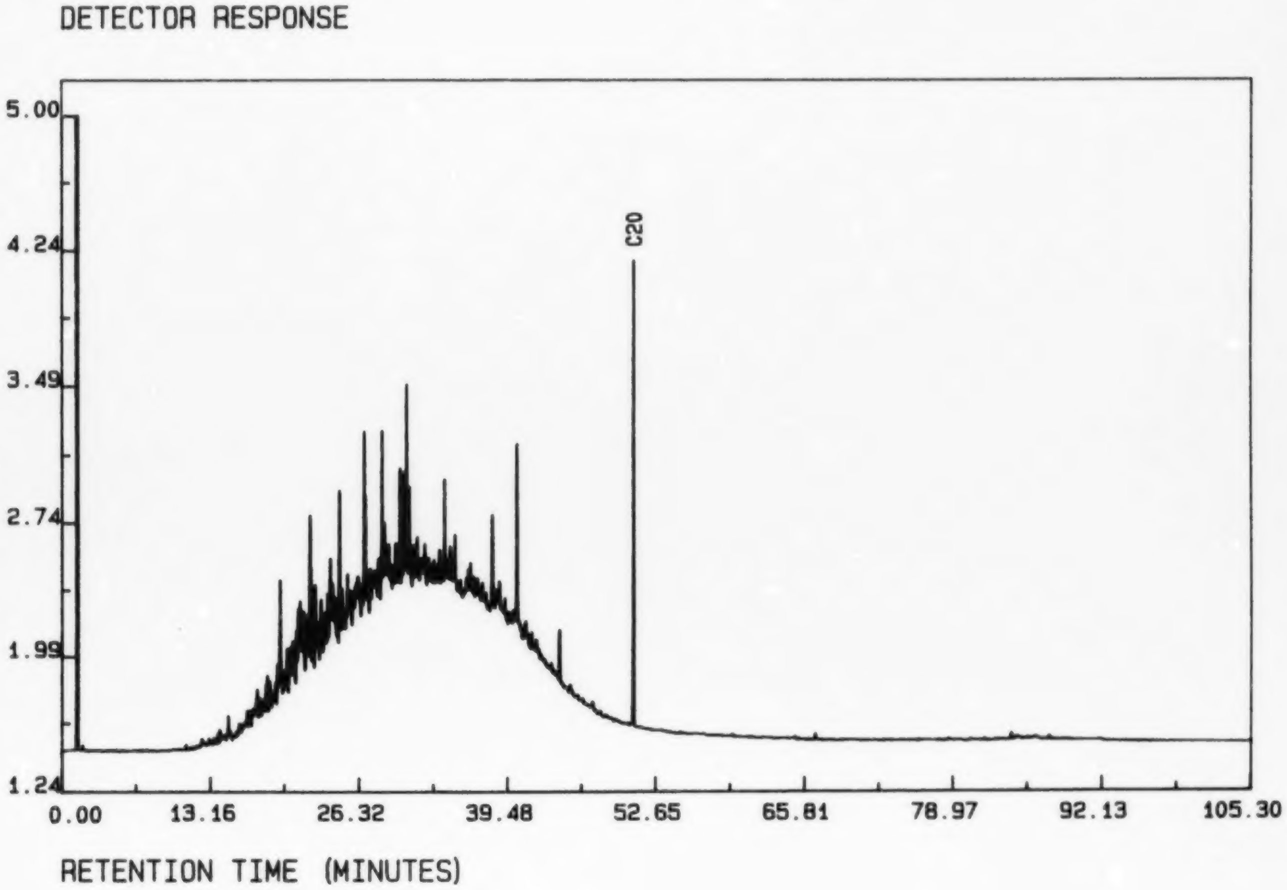

FIGURE 8

GC-FID CHROMATOGRAM OF #6 LOWER BIOREACTOR

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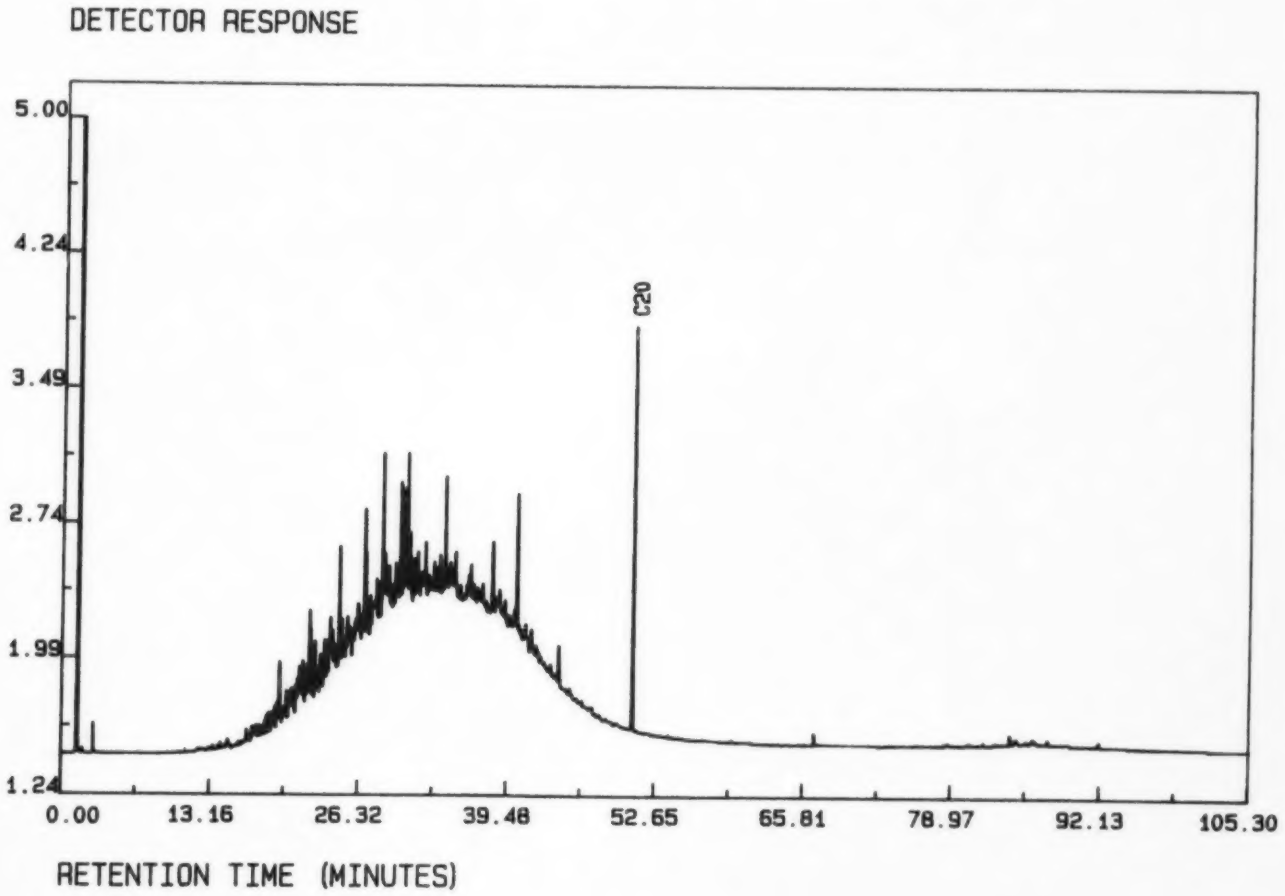

FIGURE 9

GC-FID CHROMATOGRAM OF #1 UPPER BIOREACTOR

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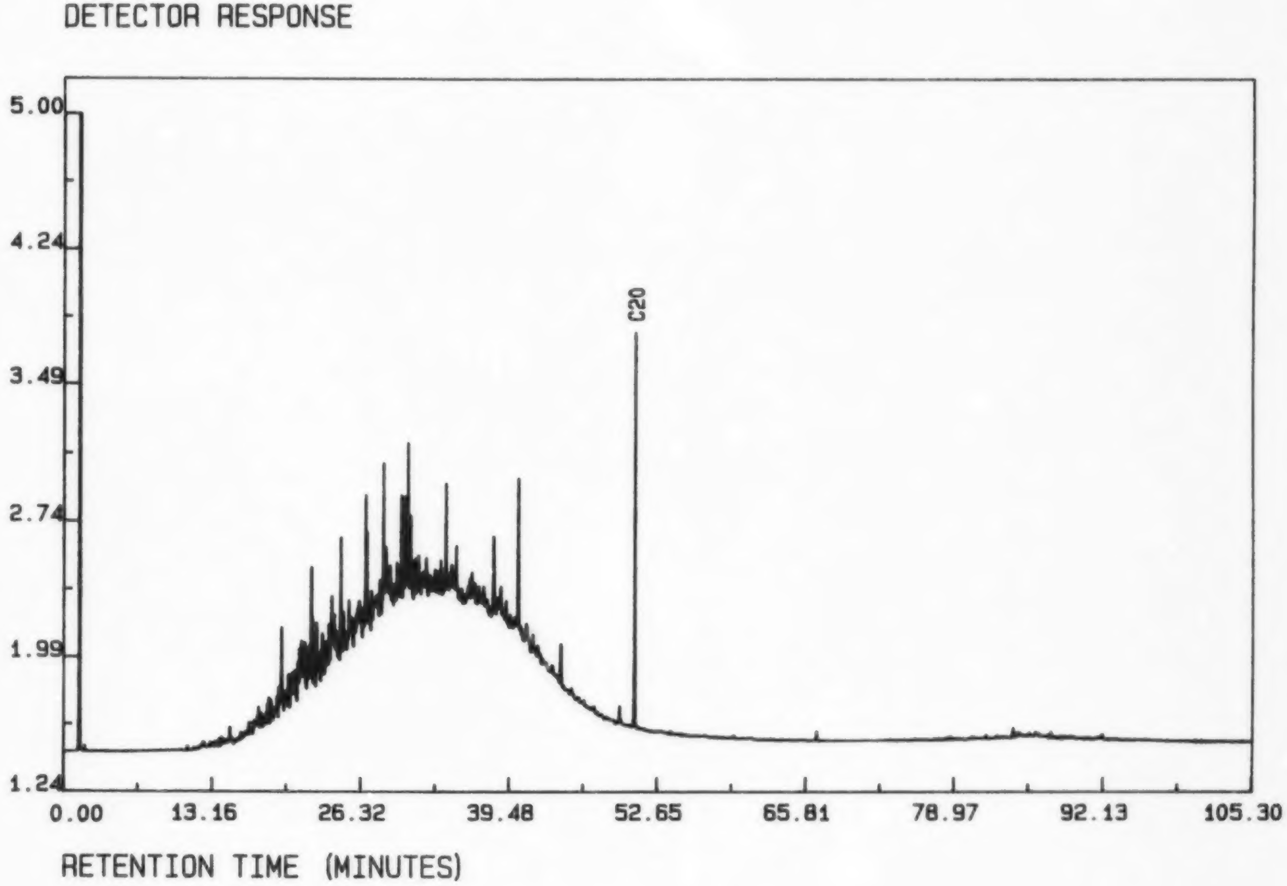

FIGURE 10

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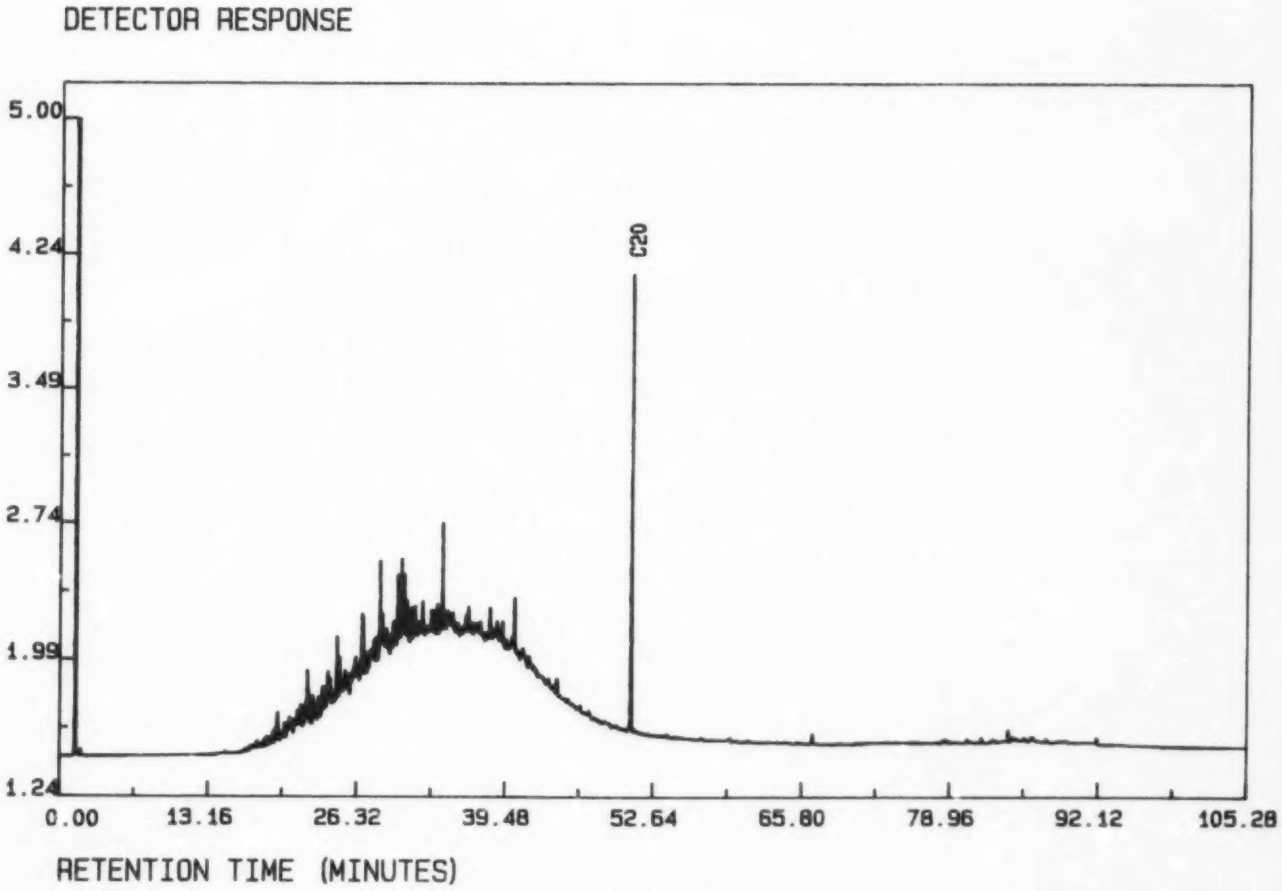

FIGURE 11

GC-FID CHROMATOGRAM OF #1 LOWER BIOREACTOR

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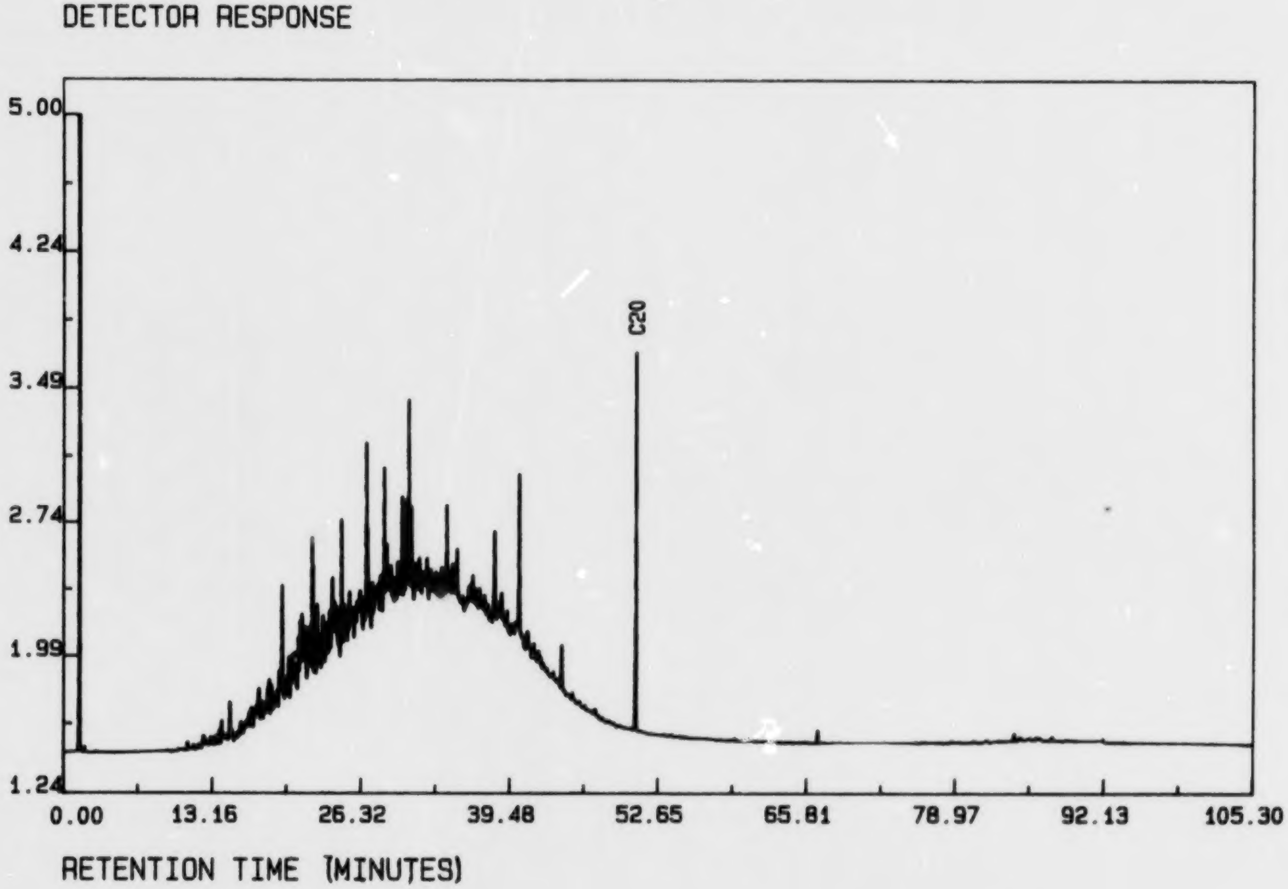

FIGURE 12

GC-FID CHROMATOGRAM OF #6 LOWER BIOREACTOR

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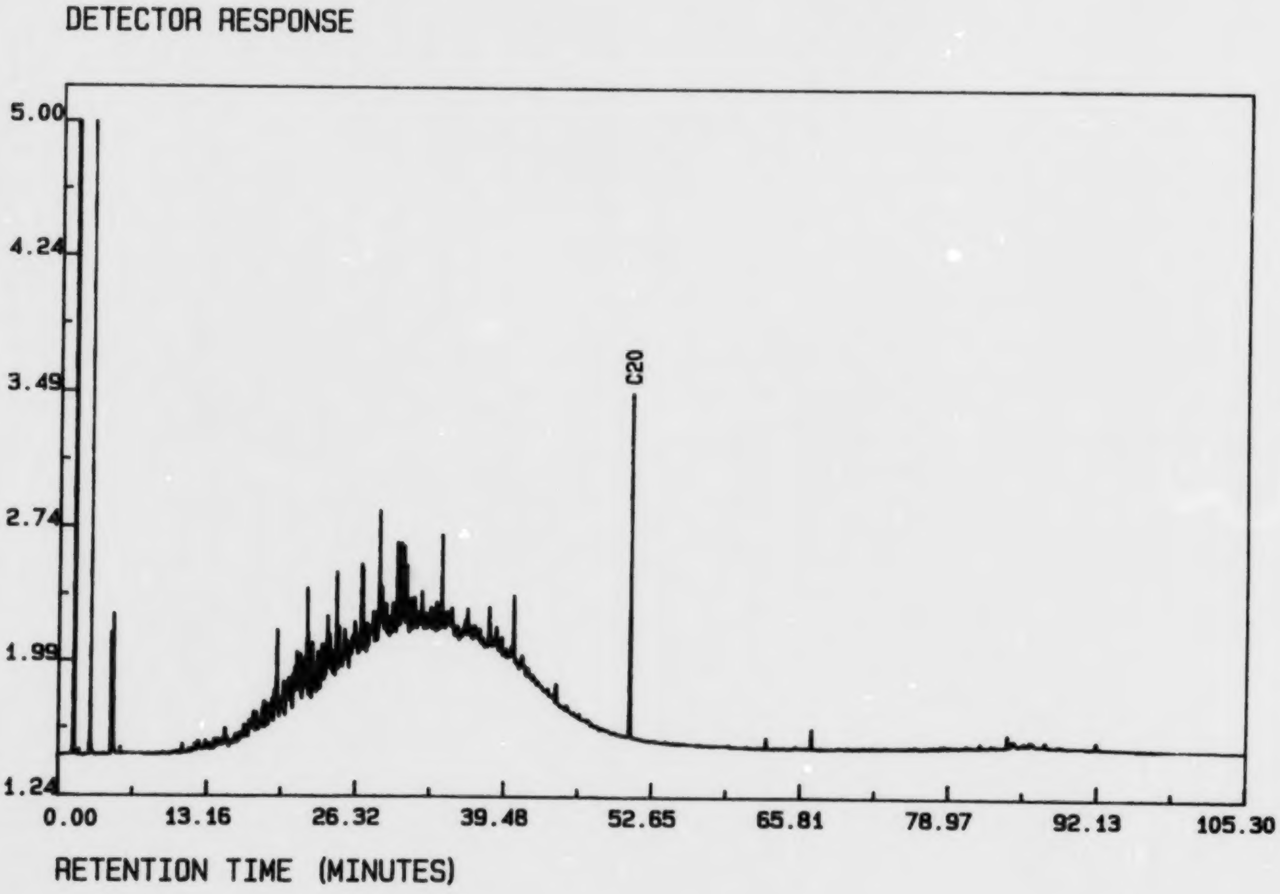

FIGURE 13
GC-FID CHROMATOGRAM OF #2 UPPER BIOREACTOR
MAY 28, 1990

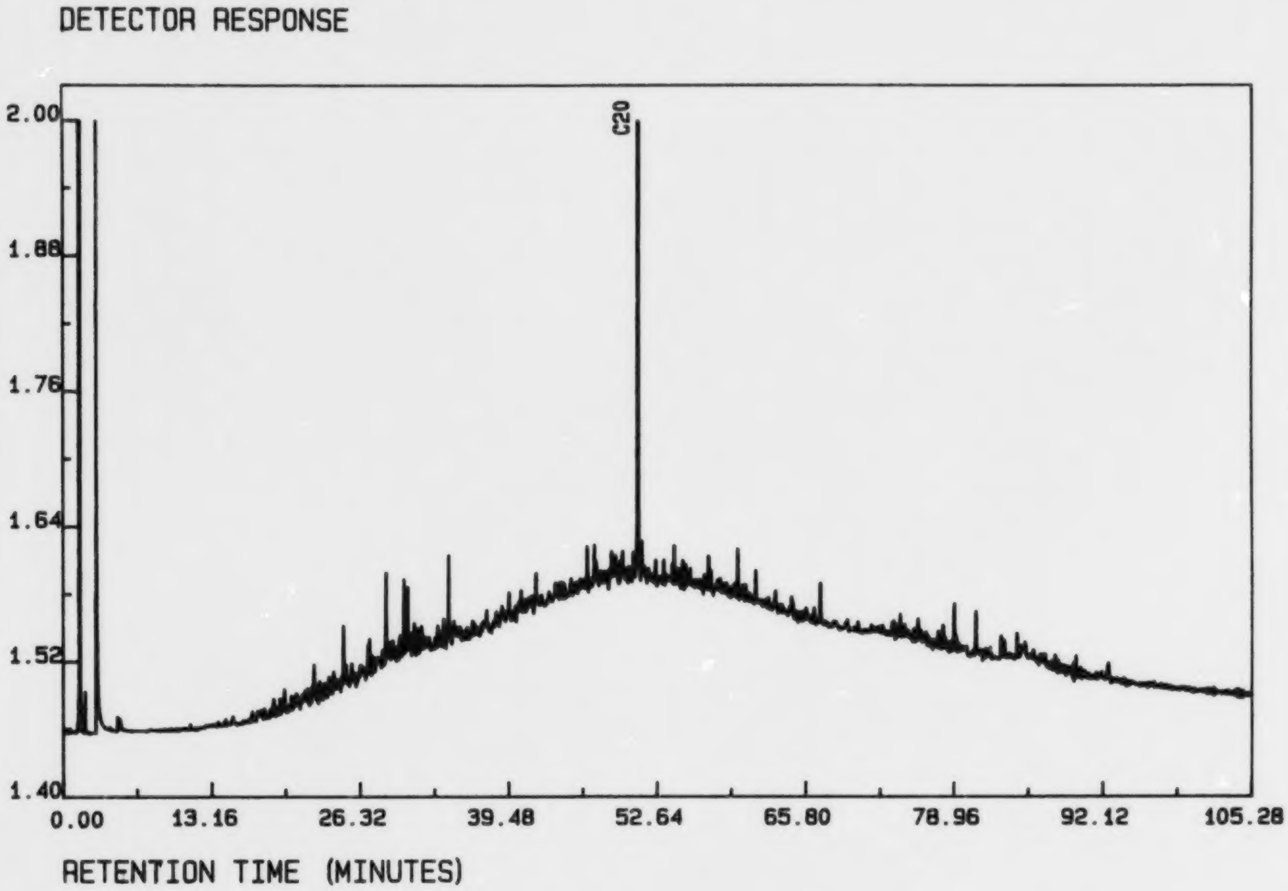

FIGURE 14

GC-FID CHROMATOGRAM OF #3 UPPER BIOREACTOR

MAY 28, 1990

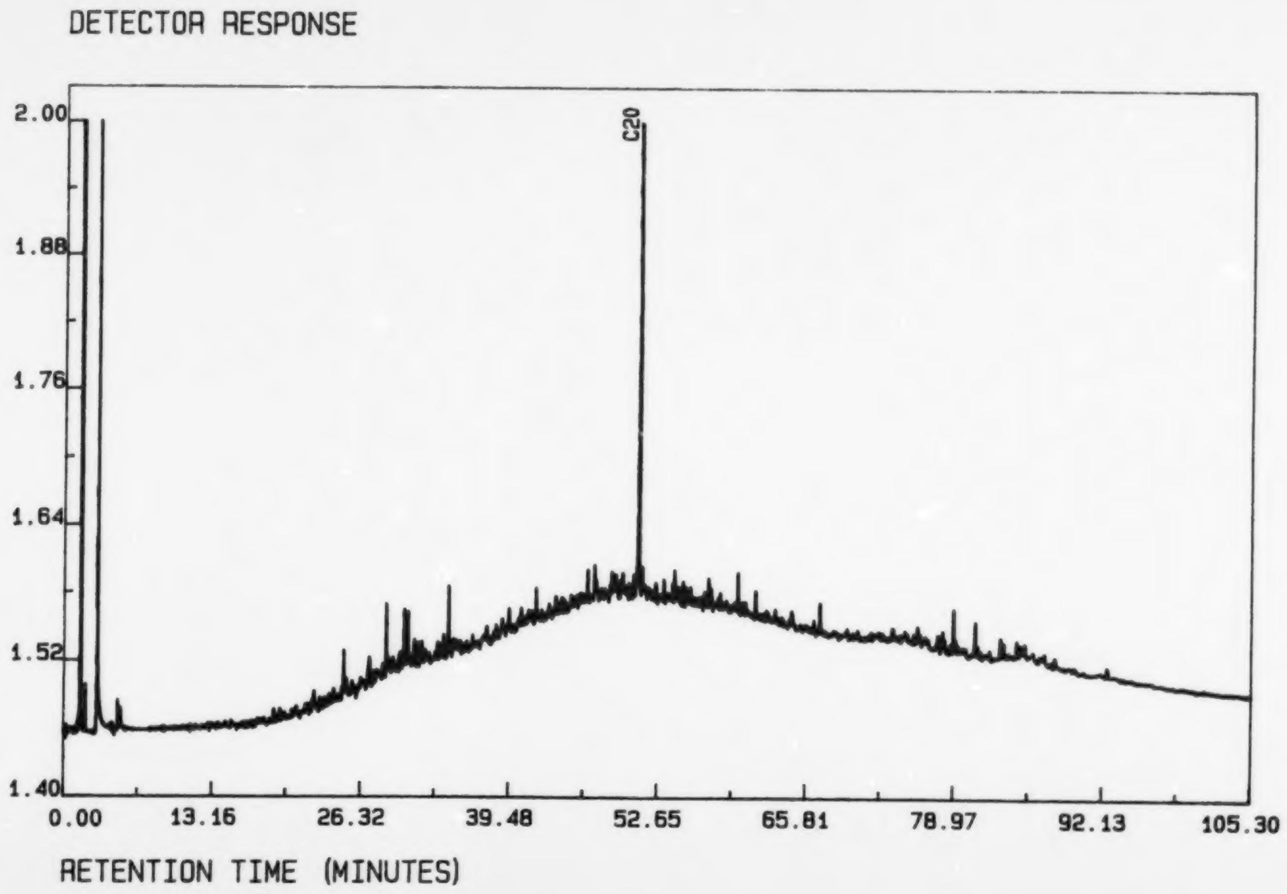

FIGURE 15

GC-FID CHROMATOGRAM OF #4 UPPER BIOREACTOR

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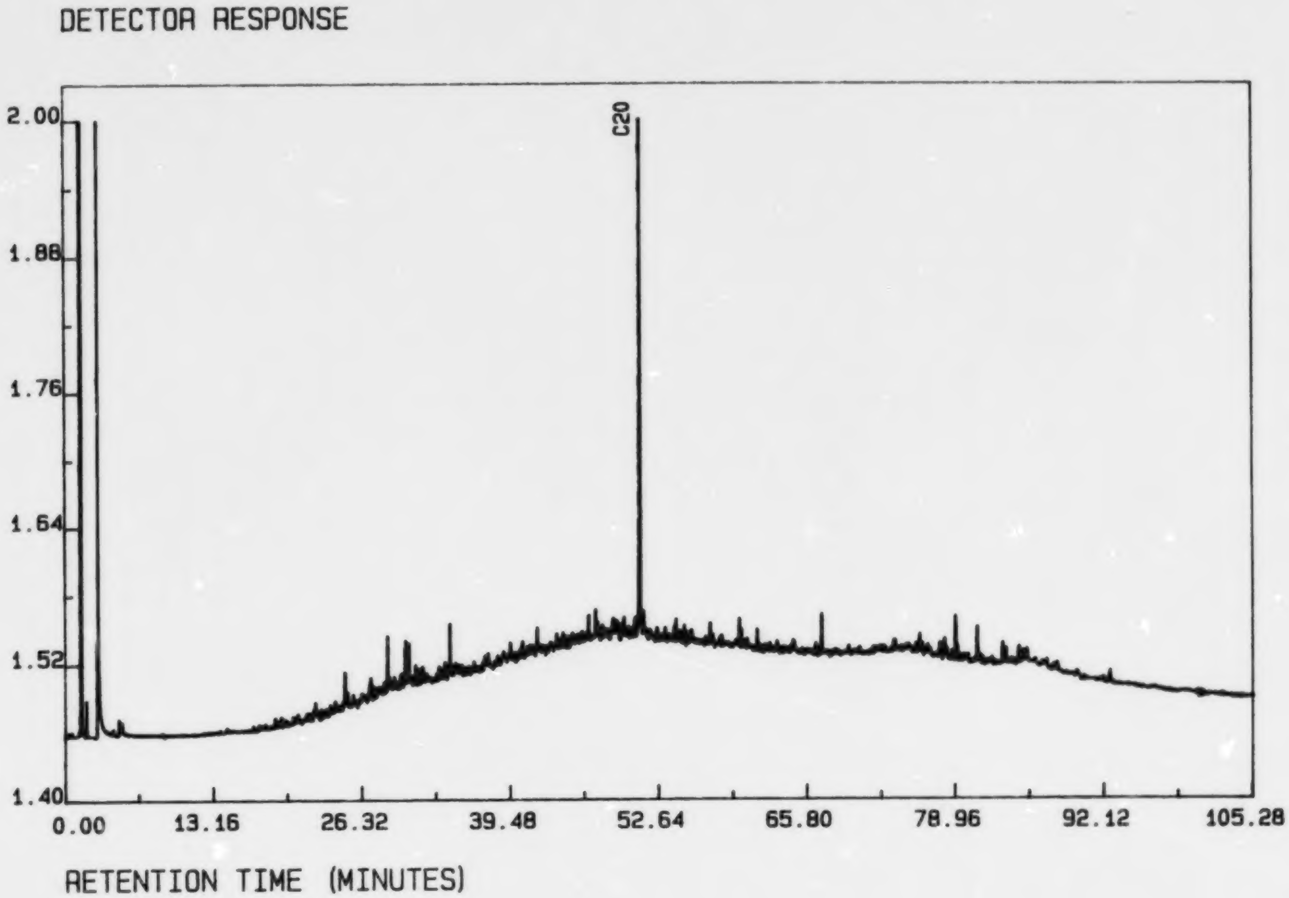

FIGURE 16

GC-FID CHROMATOGRAM OF #5 UPPER BIOREACTOR

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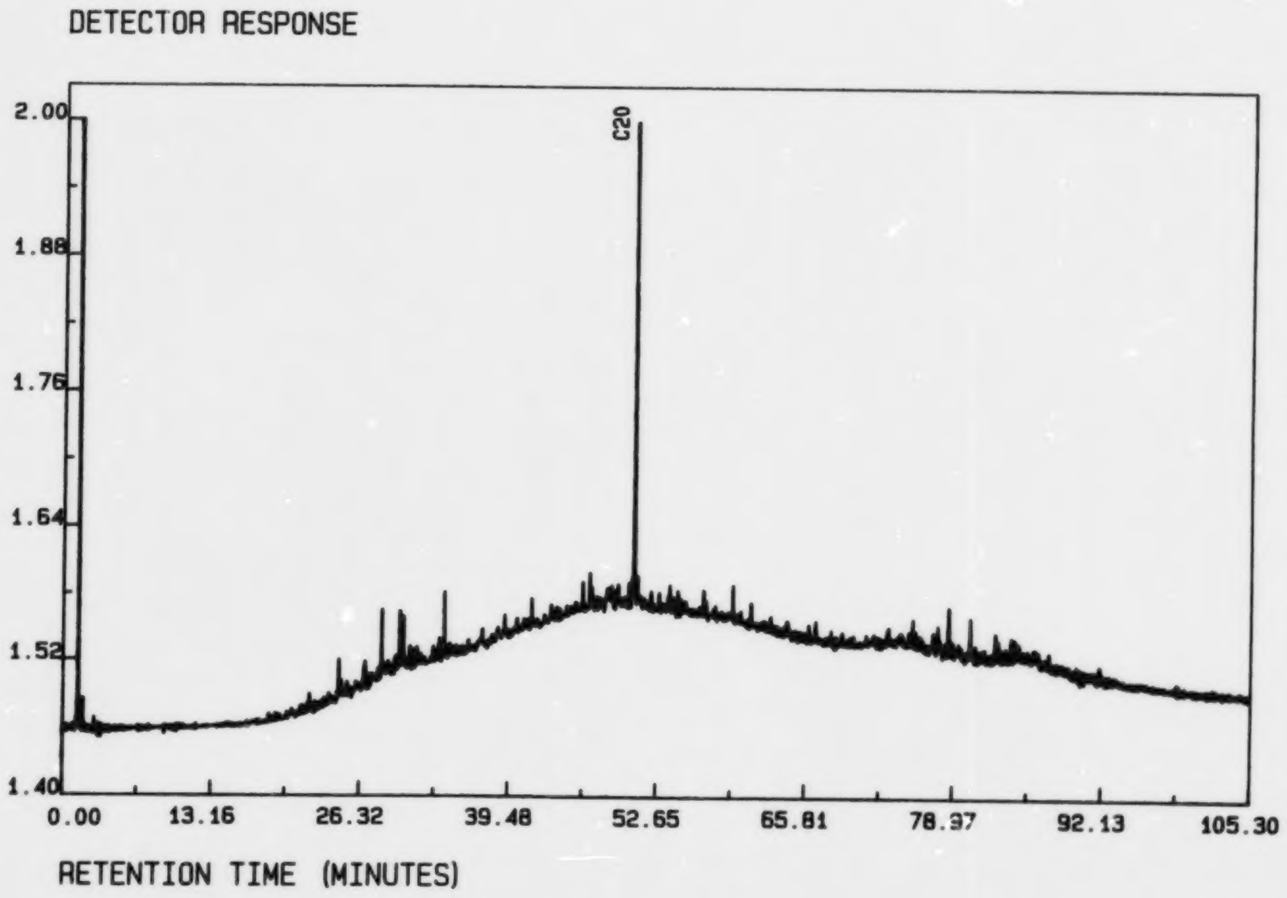

FIGURE 17
GC-FID CHROMATOGRAM OF #2 LOWER BIOREACTOR
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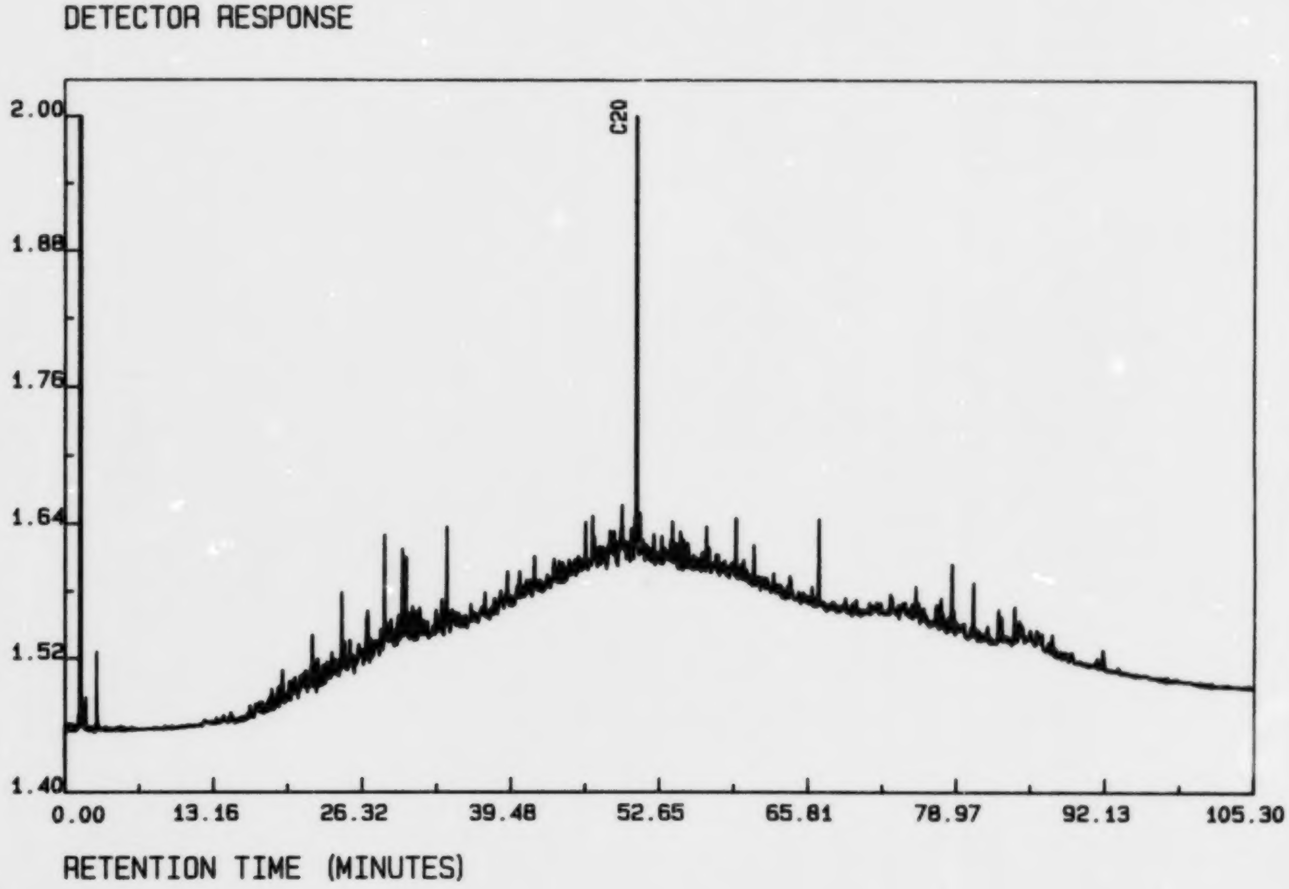

FIGURE 18

GC-FID CHROMATOGRAM OF #3 LOWER BIOREACTOR

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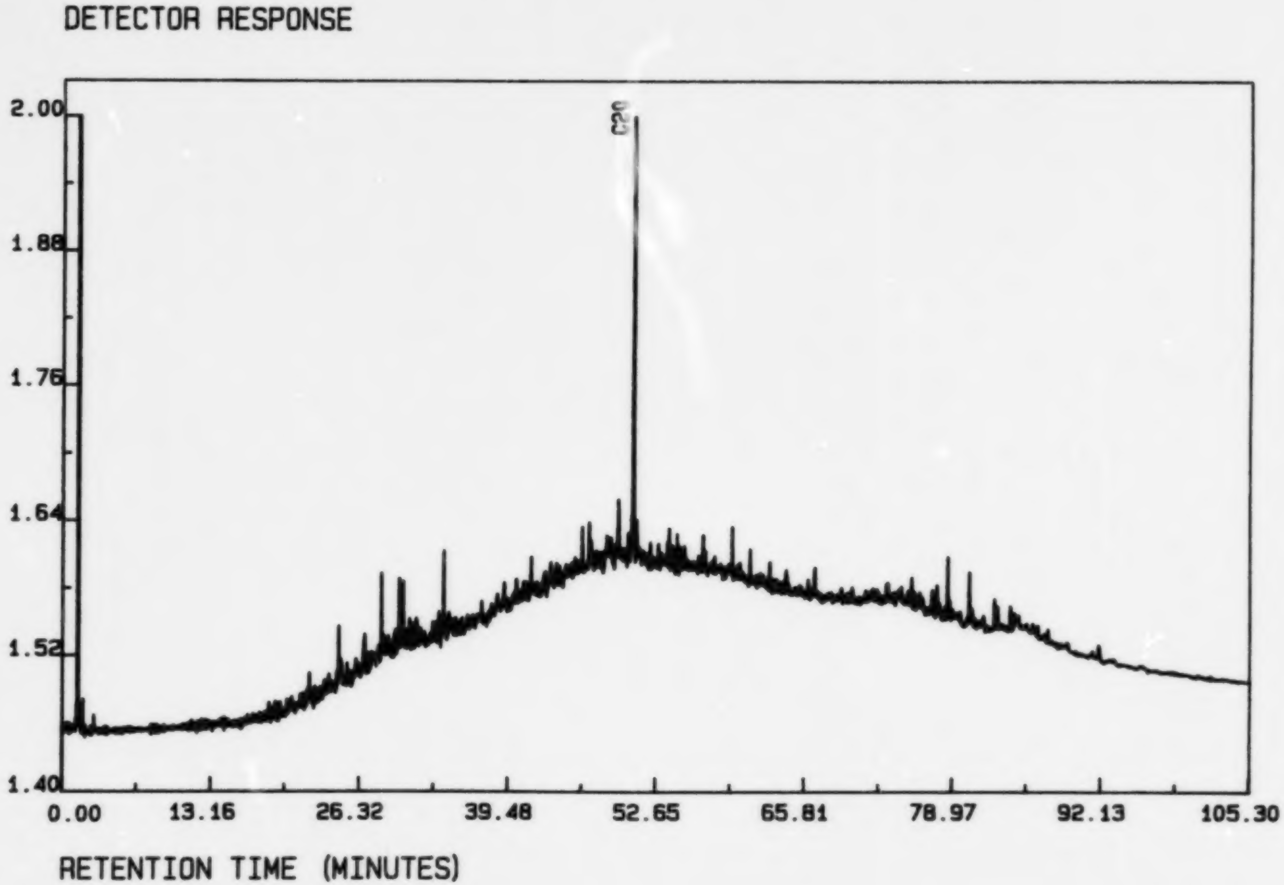

FIGURE 19

GC-FID CHROMATOGRAM OF #4 LOWER BIOREACTOR

MAY 28, 1990

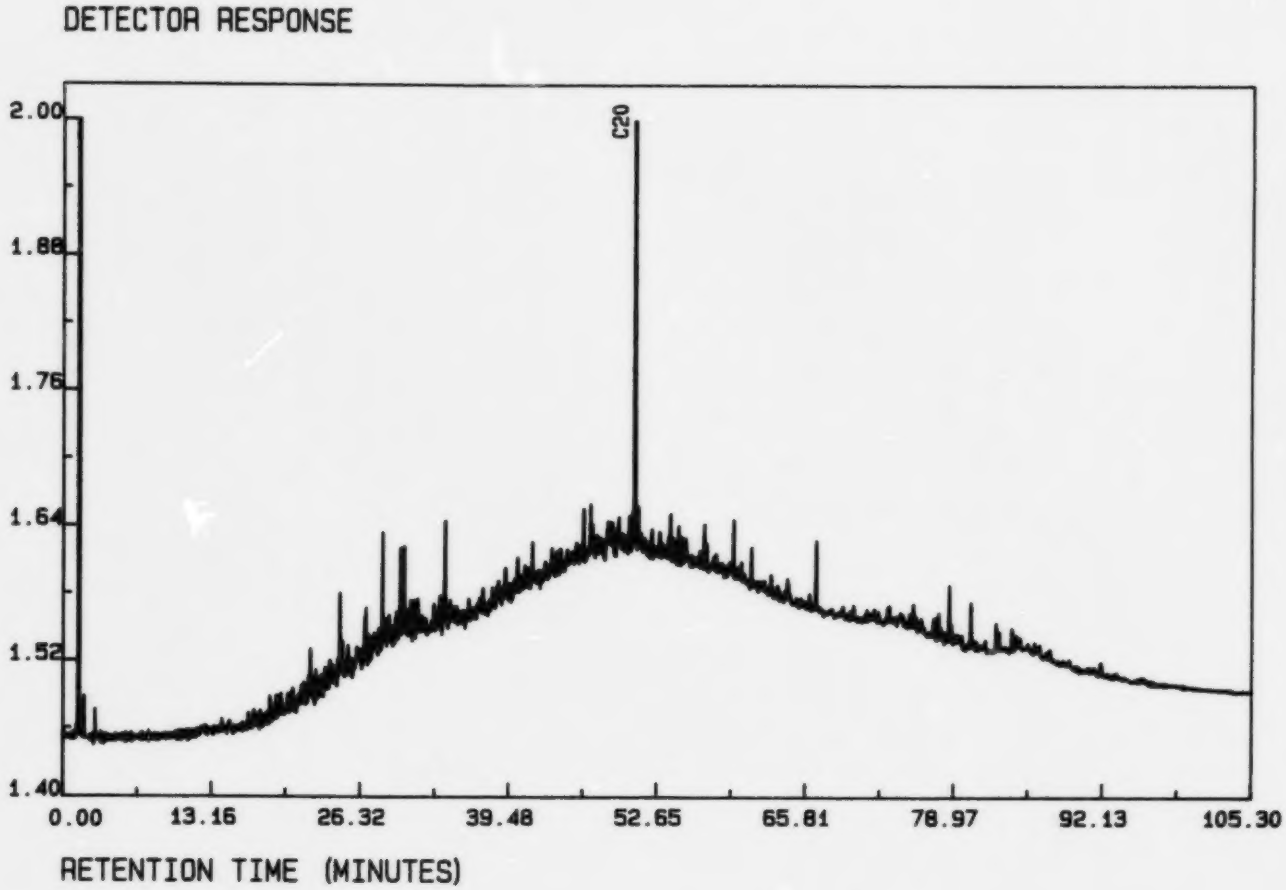

FIGURE 20

GC-FID CHROMATOGRAM OF #5 LOWER BIOREACTOR

MAY 28, 1990

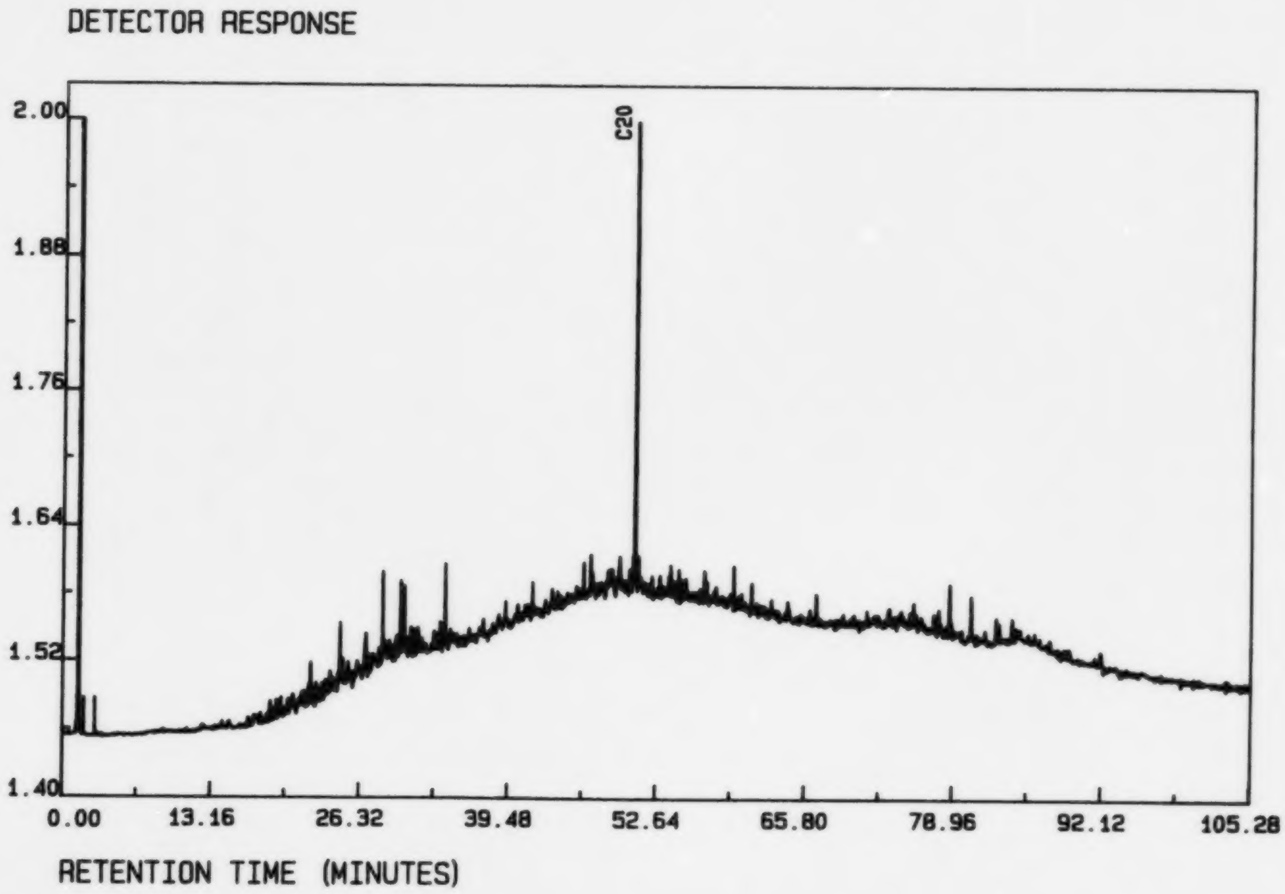

FIGURE 21

GC-FID CHROMATOGRAM OF #2 UPPER BIOREACTOR

JUNE 13, 1990

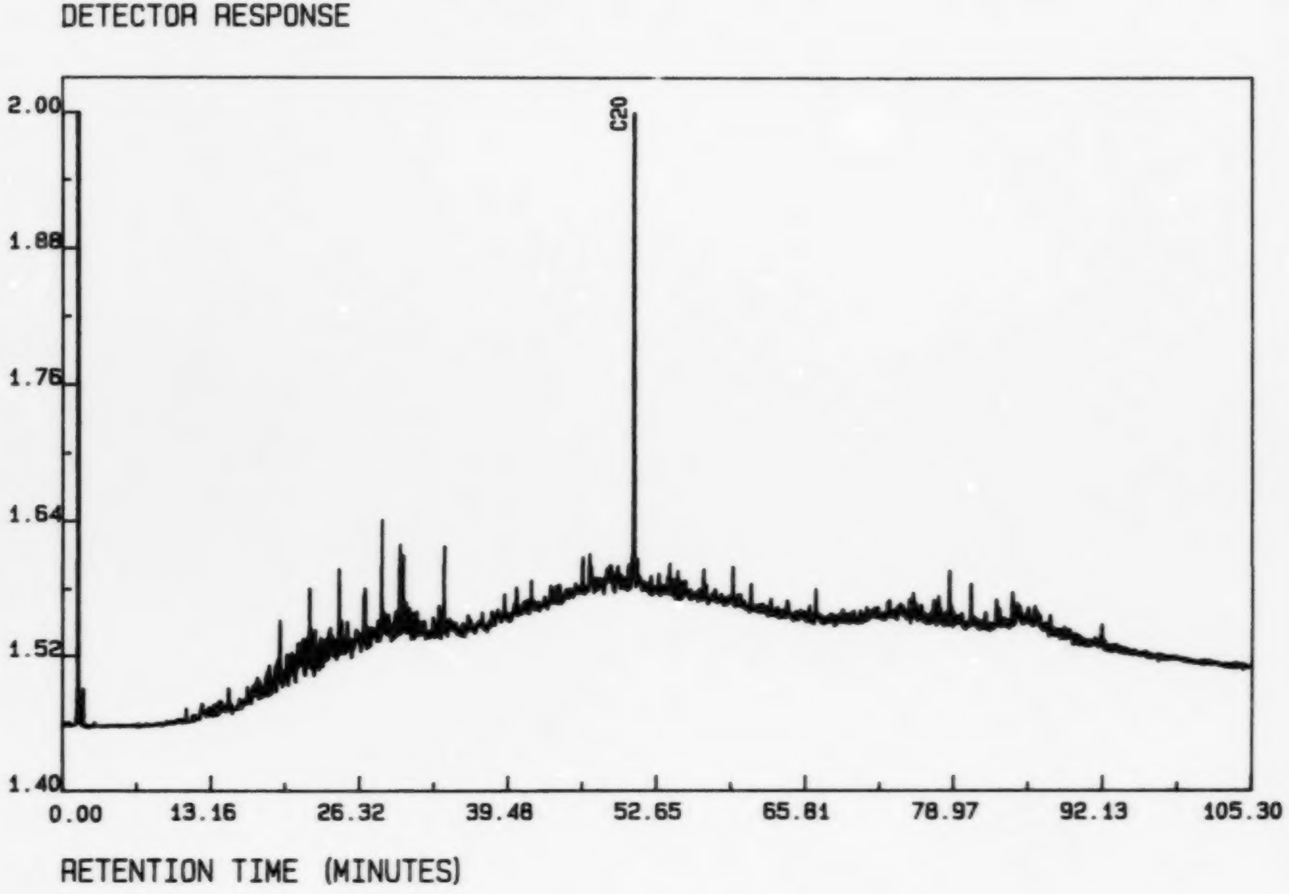

FIGURE 22

GC-FID CHROMATOGRAM OF #3 UPPER BIOREACTOR

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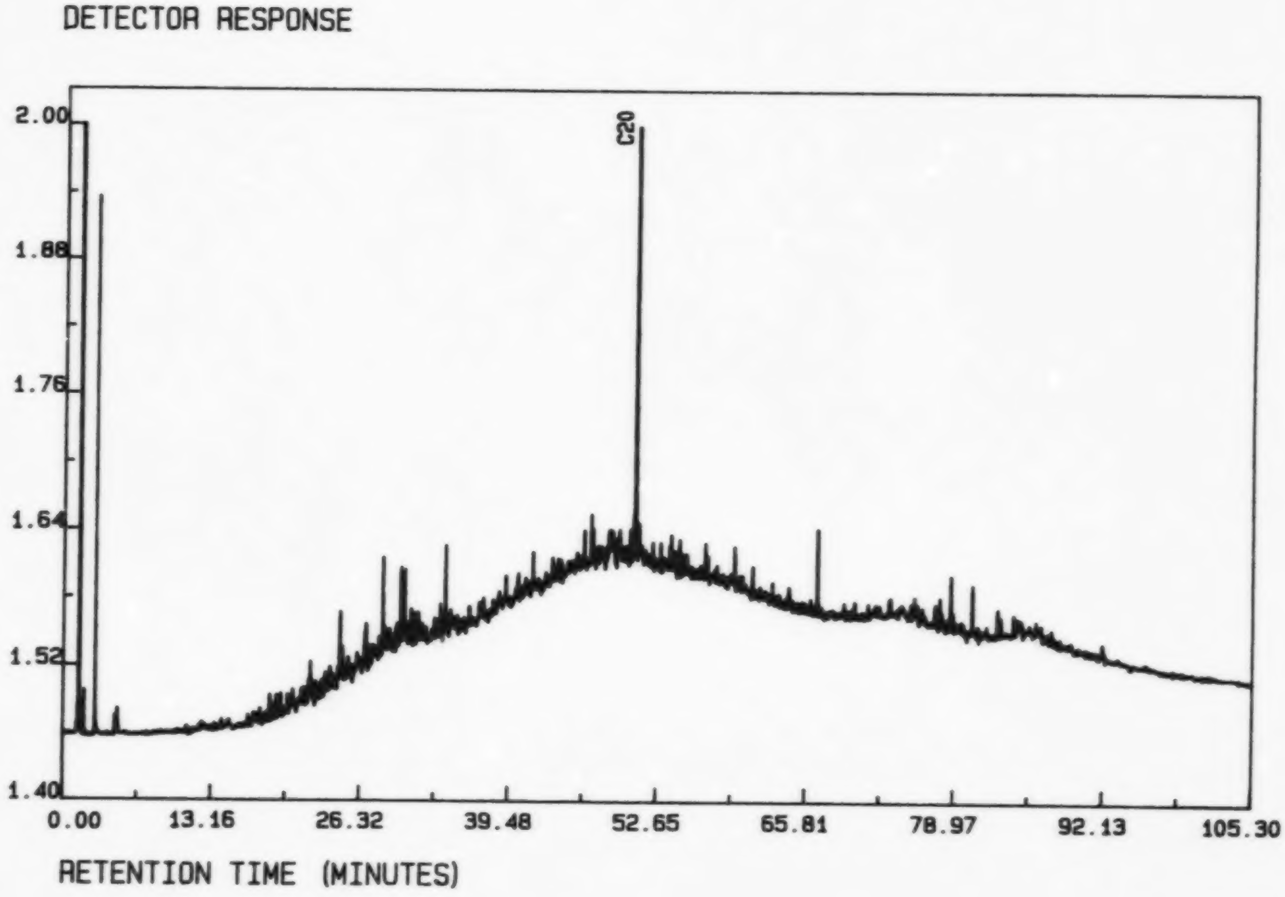

FIGURE 23
GC-FID CHROMATOGRAM OF #4 UPPER BIOREACTOR
JUNE 13, 1990

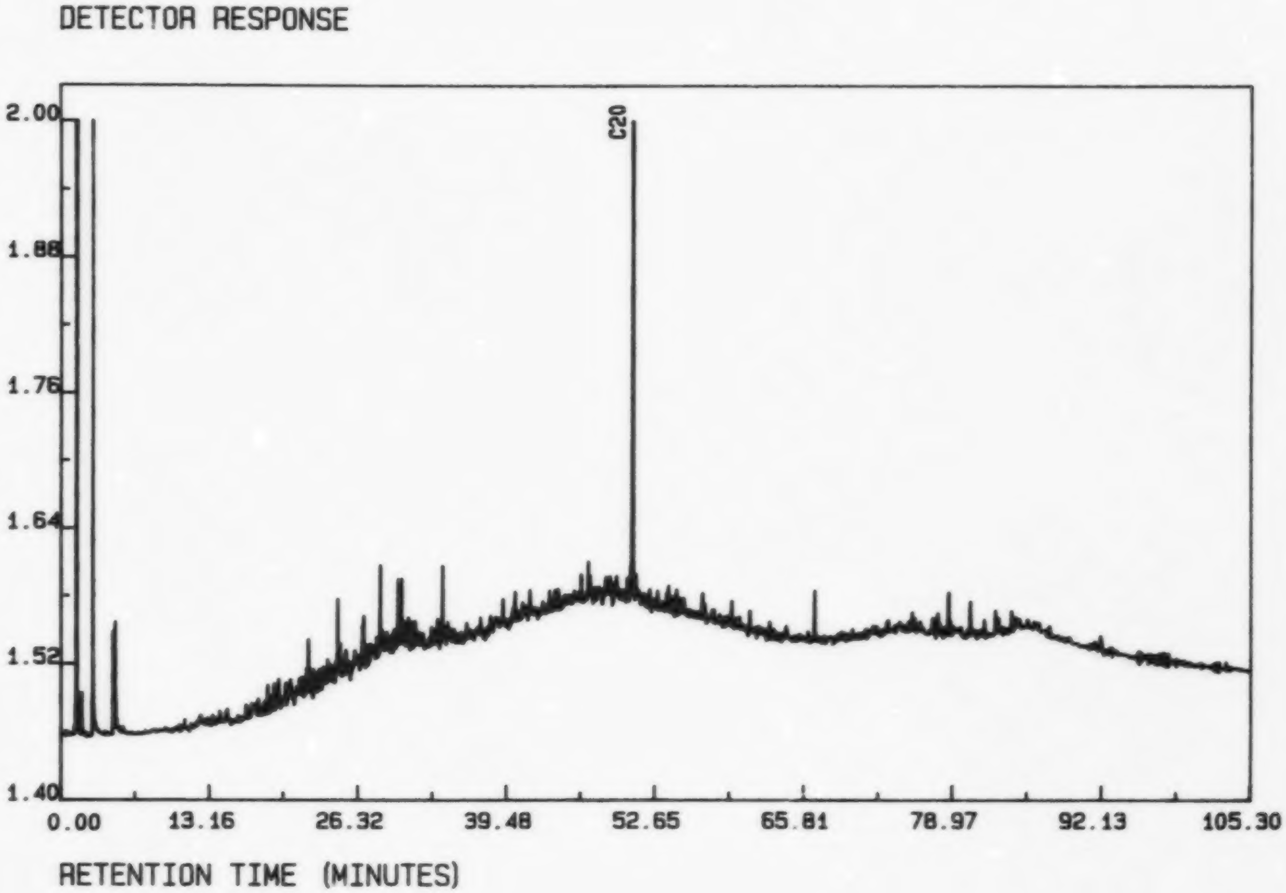

FIGURE 24

GC-FID CHROMATOGRAM OF #5 UPPER BIOREACTOR

JUNE 13, 1990

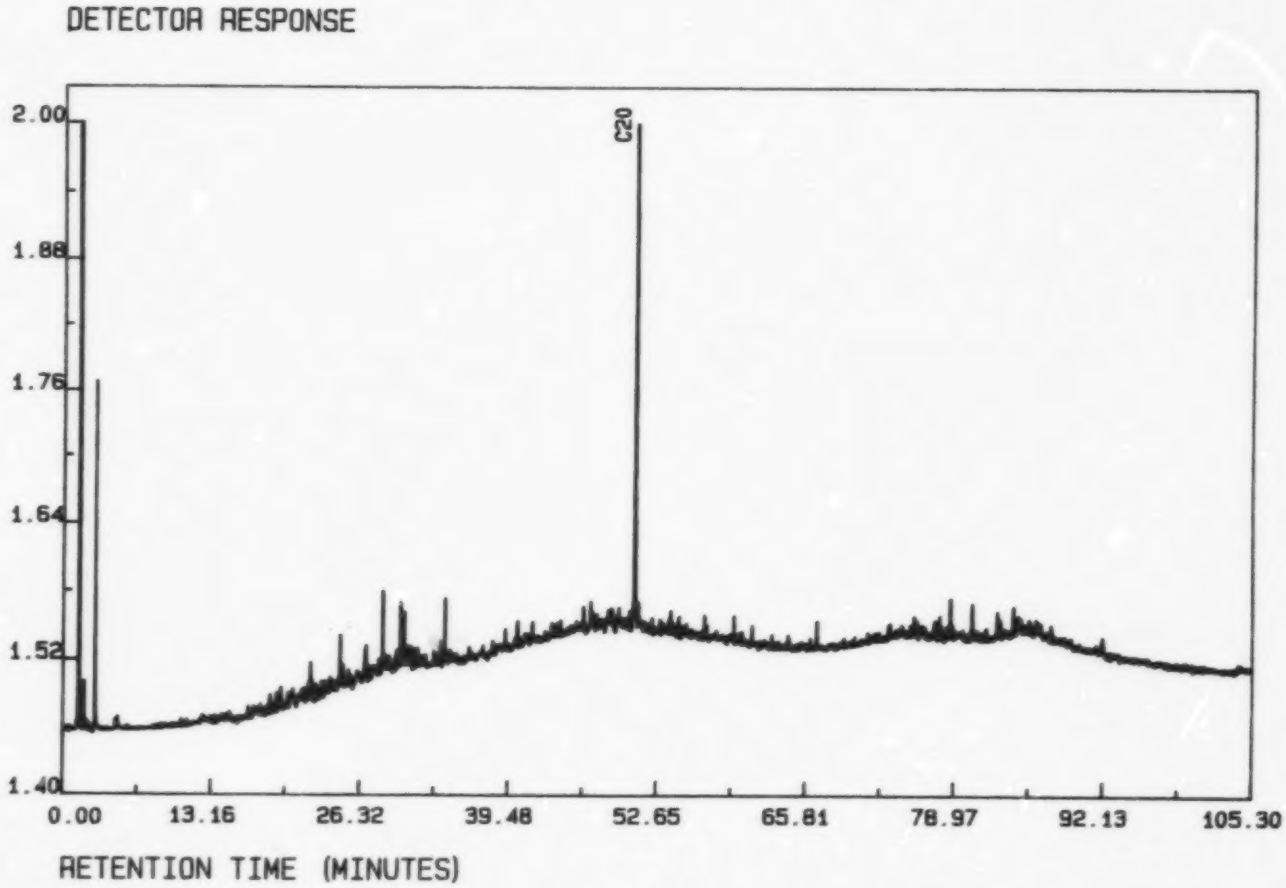

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FIGURE 25
GC-FID CHROMATOGRAM OF #2 LOWER BIOREACTOR
JUNE 13, 1990

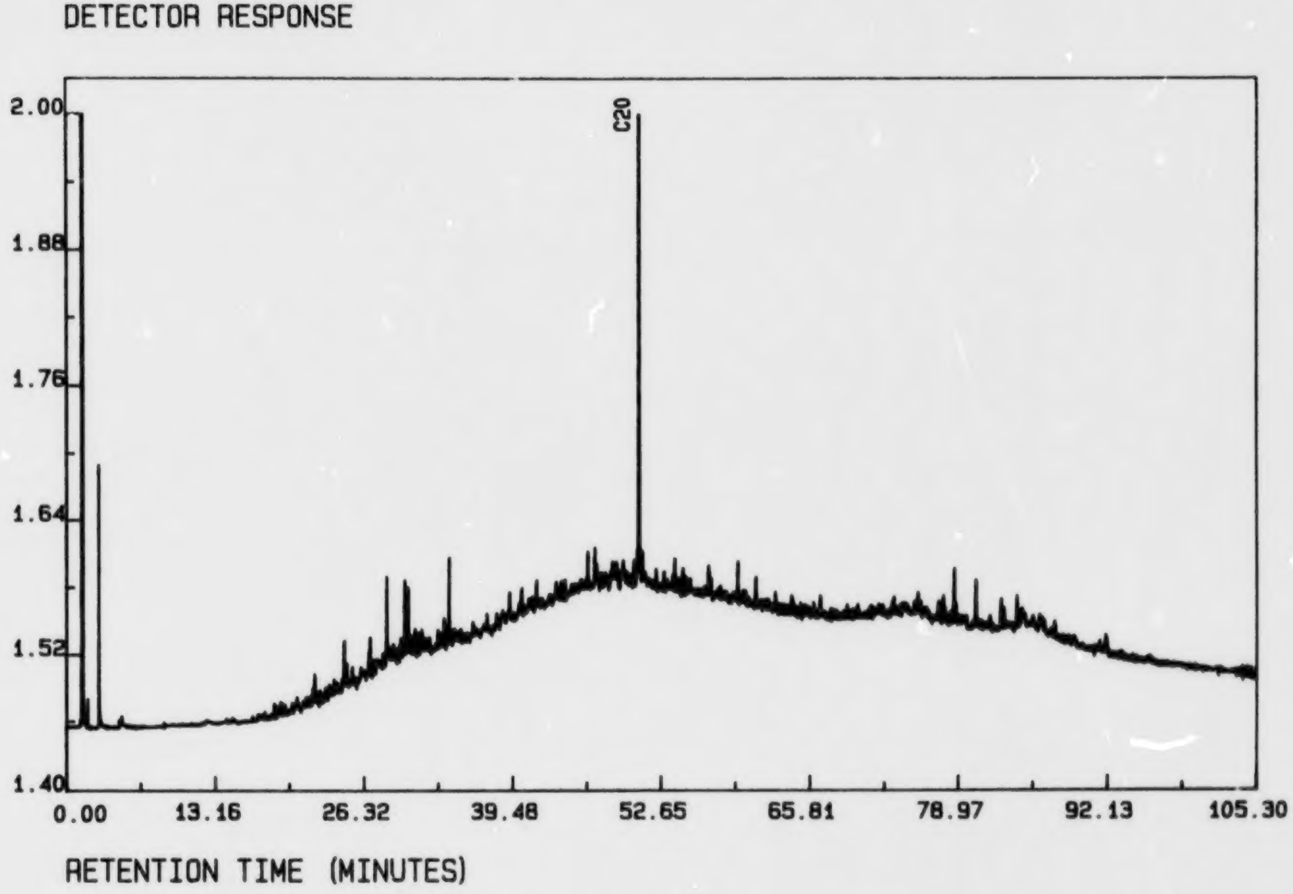


FIGURE 26

GC-FID CHROMATOGRAM OF #3 LOWER BIOREACTOR

JUNE 13, 1990

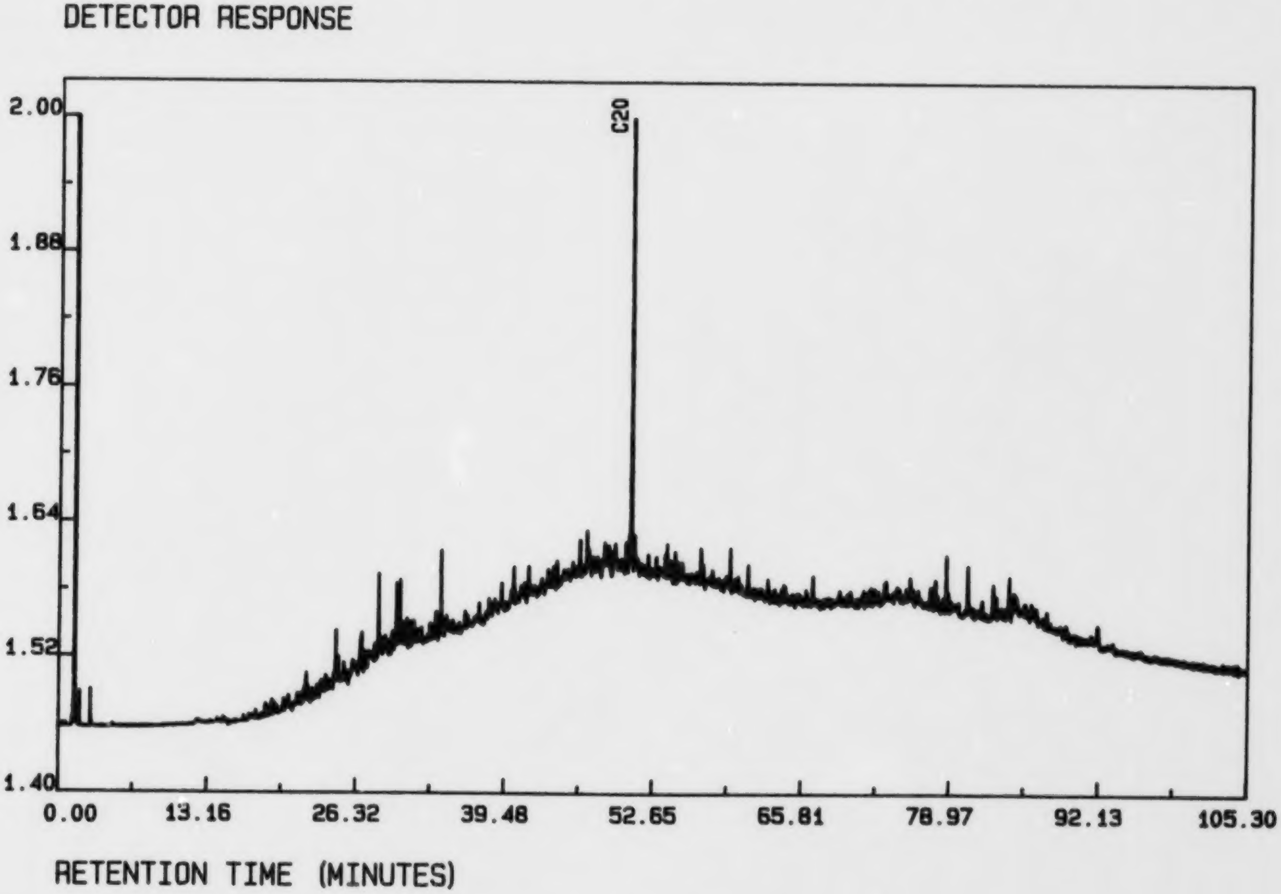


FIGURE 27

GC-FID CHROMATOGRAM OF #4 LOWER BIOREACTOR

JUNE 13, 1990

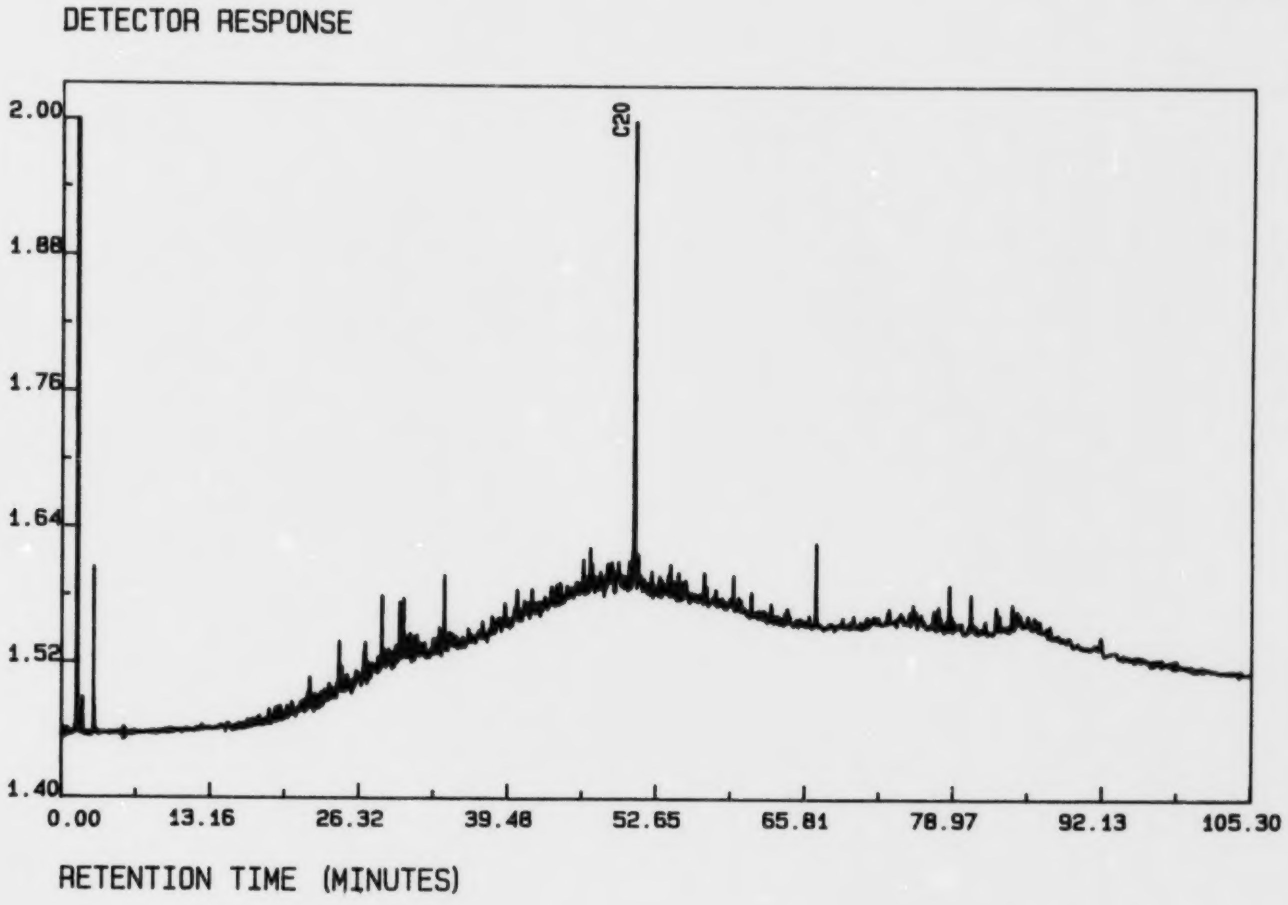


FIGURE 28

GC-FID CHROMATOGRAM OF #5 LOWER BIOREACTOR

JUNE 13, 1990

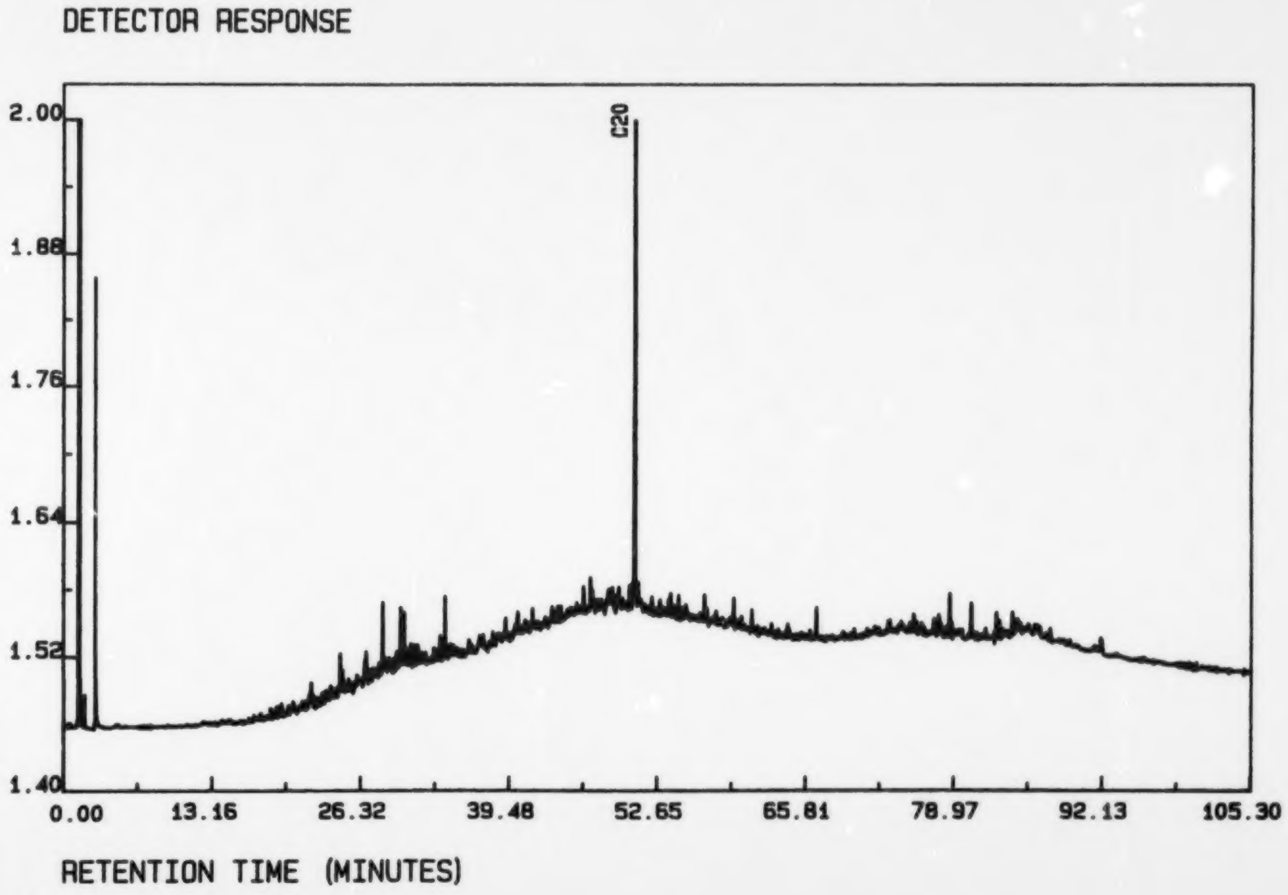


FIGURE 29

GC-FID CHROMATOGRAM OF #2 UPPER BIOREACTOR

JULY 19, 1990

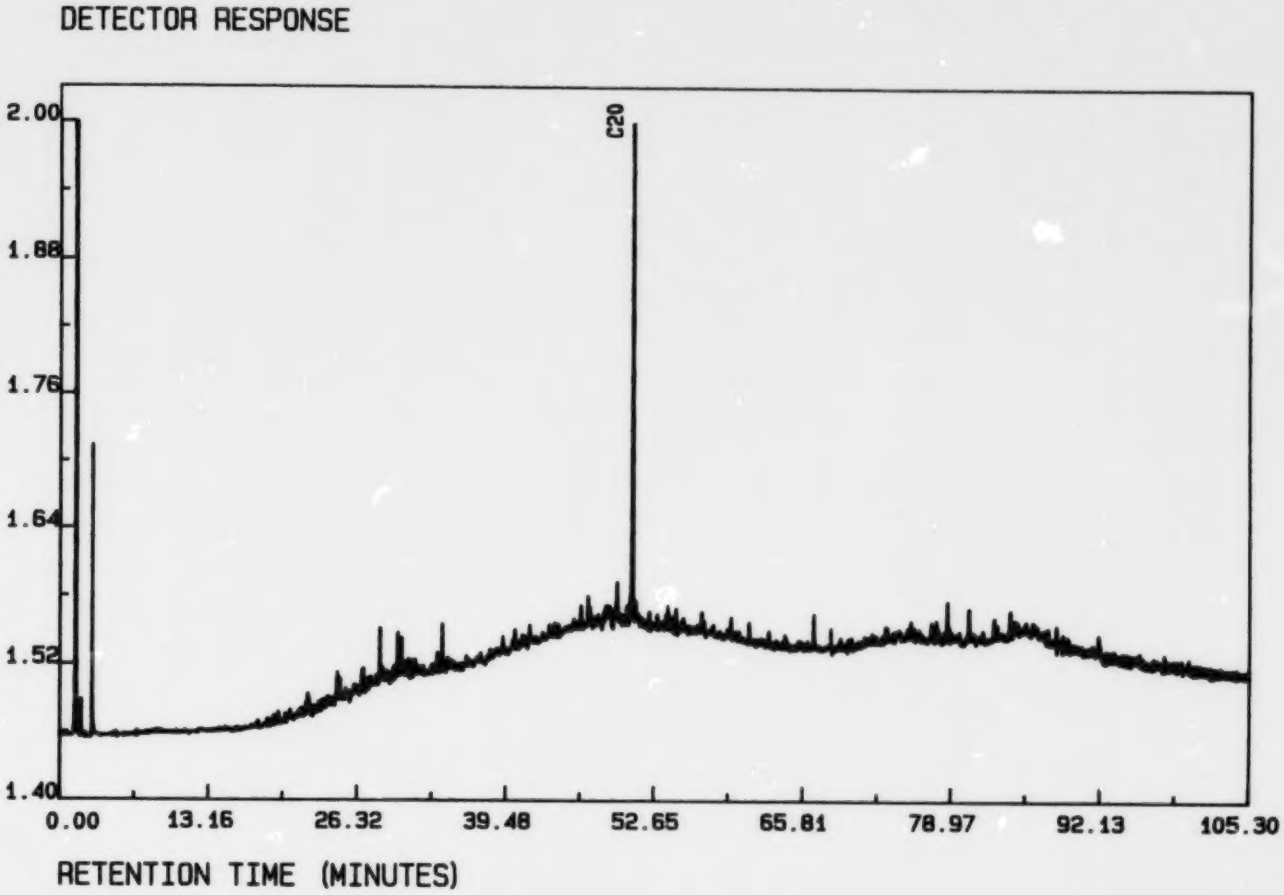


FIGURE 30

GC-FID CHROMATOGRAM OF #3 UPPER BIOREACTOR

JULY 19, 1990

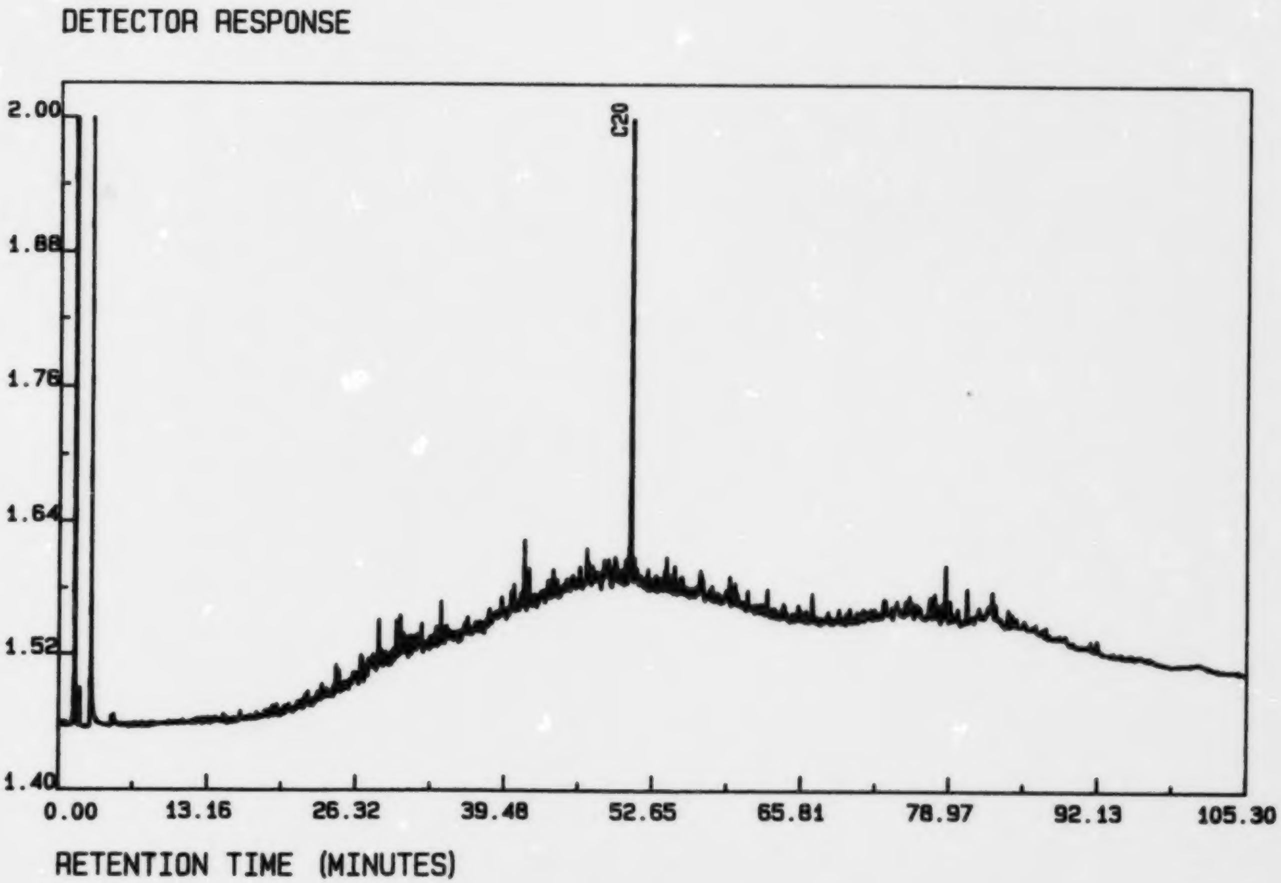


FIGURE 31
GC-FID CHROMATOGRAM OF #4 UPPER BIOREACTOR
JULY 19, 1990

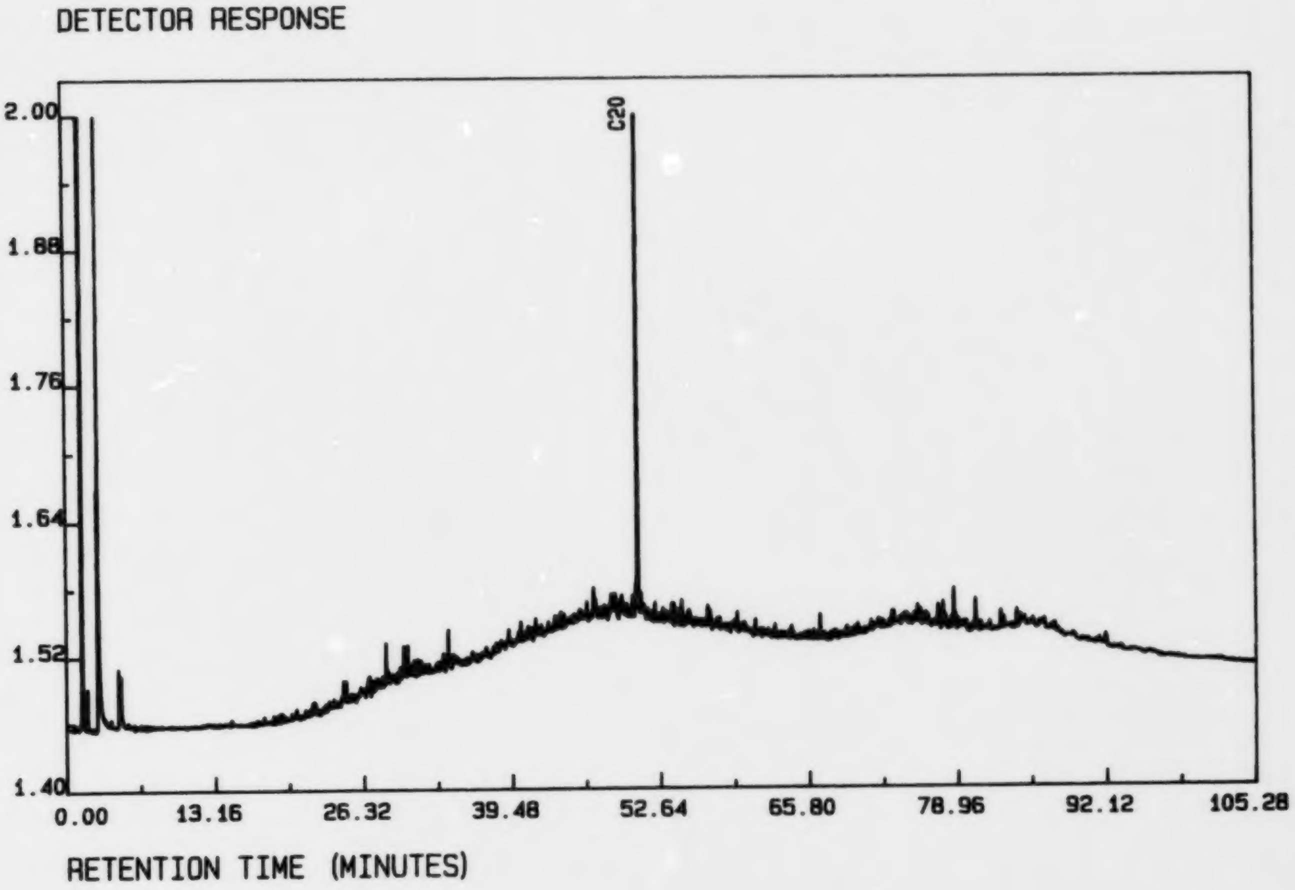


FIGURE 32

GC-FID CHROMATOGRAM OF #5 UPPER BIOREACTOR

JULY 19, 1990

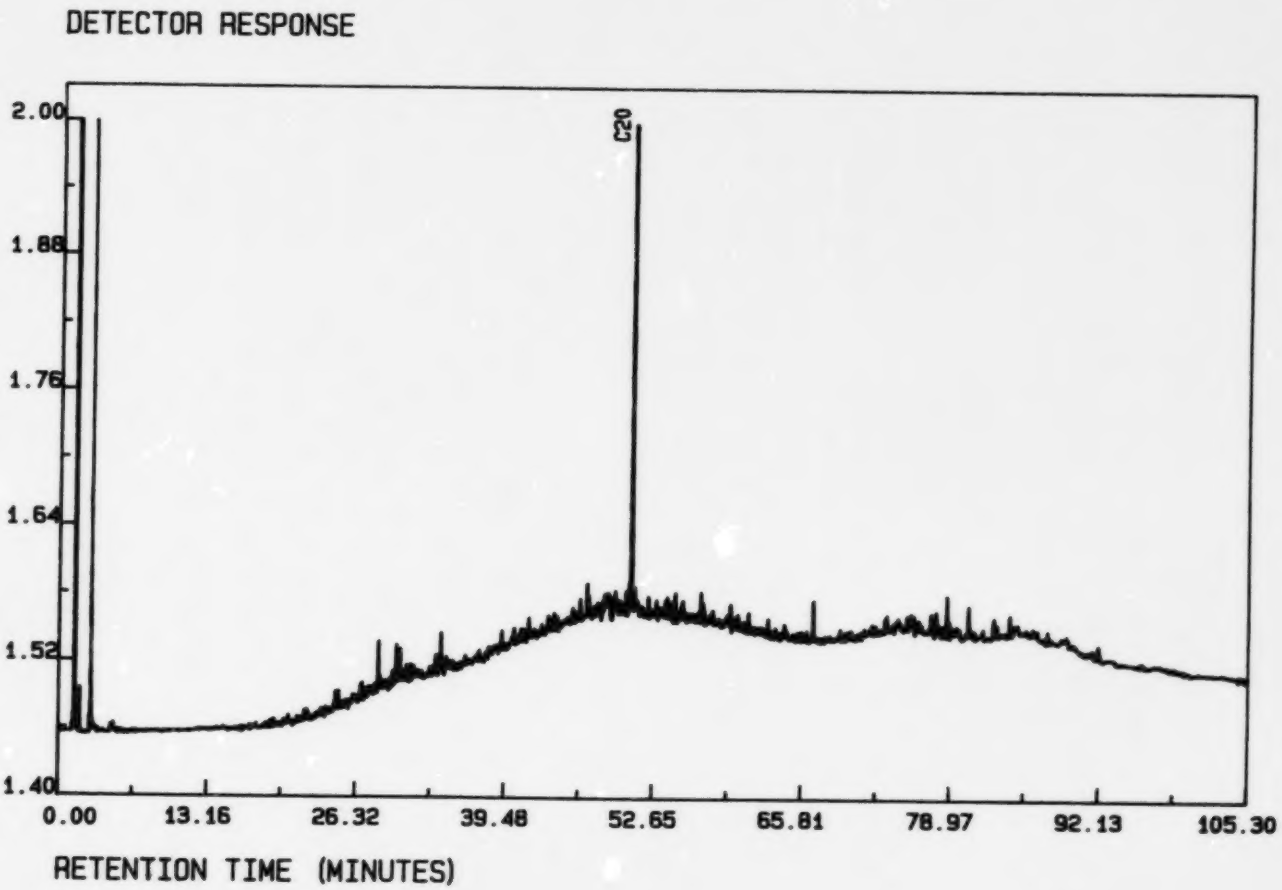


FIGURE 33

GC-FID CHROMATOGRAM OF #2 LOWER BIOREACTOR

JULY 19, 1990

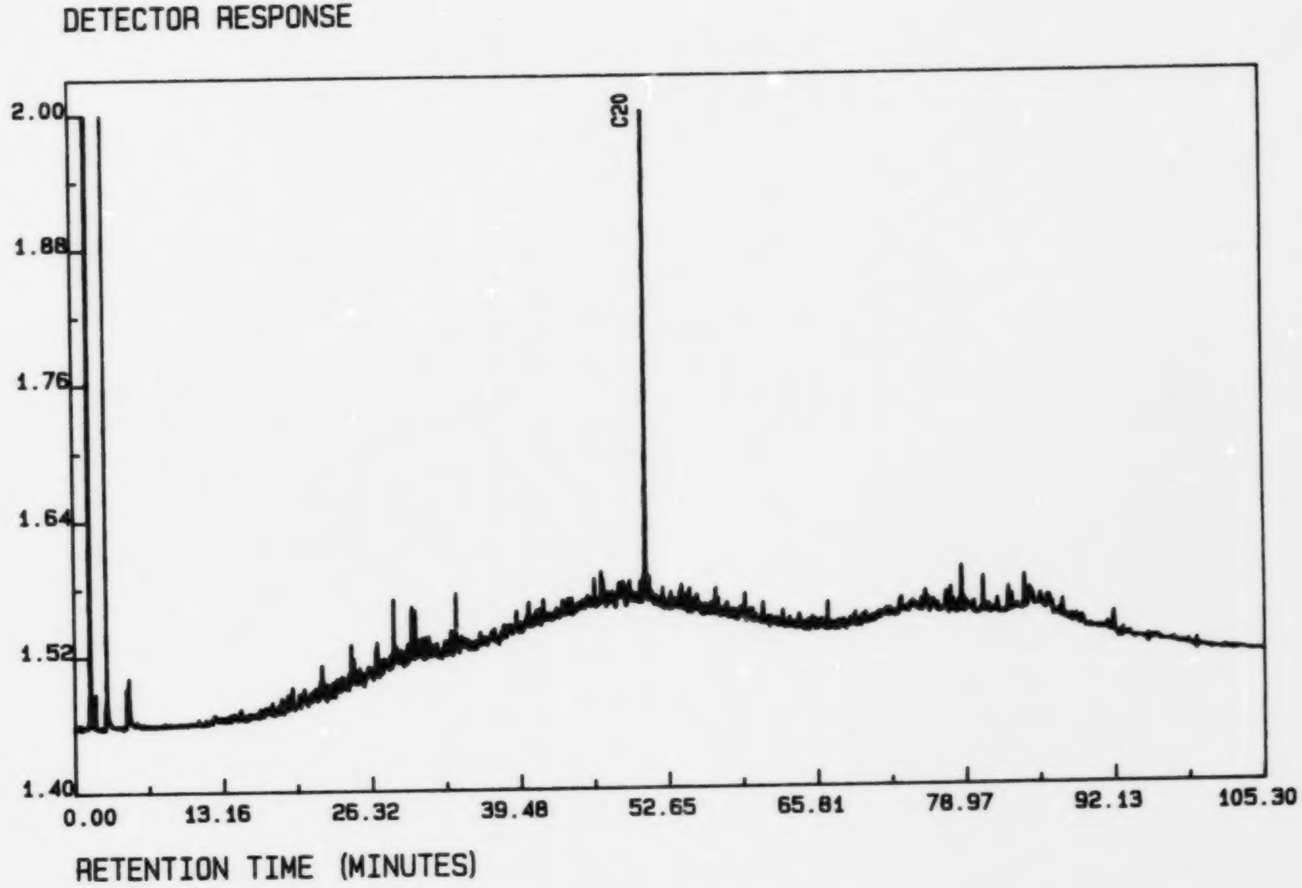


FIGURE 34

GC-FID CHROMATOGRAM OF #3 LOWER BIOREACTOR

JULY 19, 1990

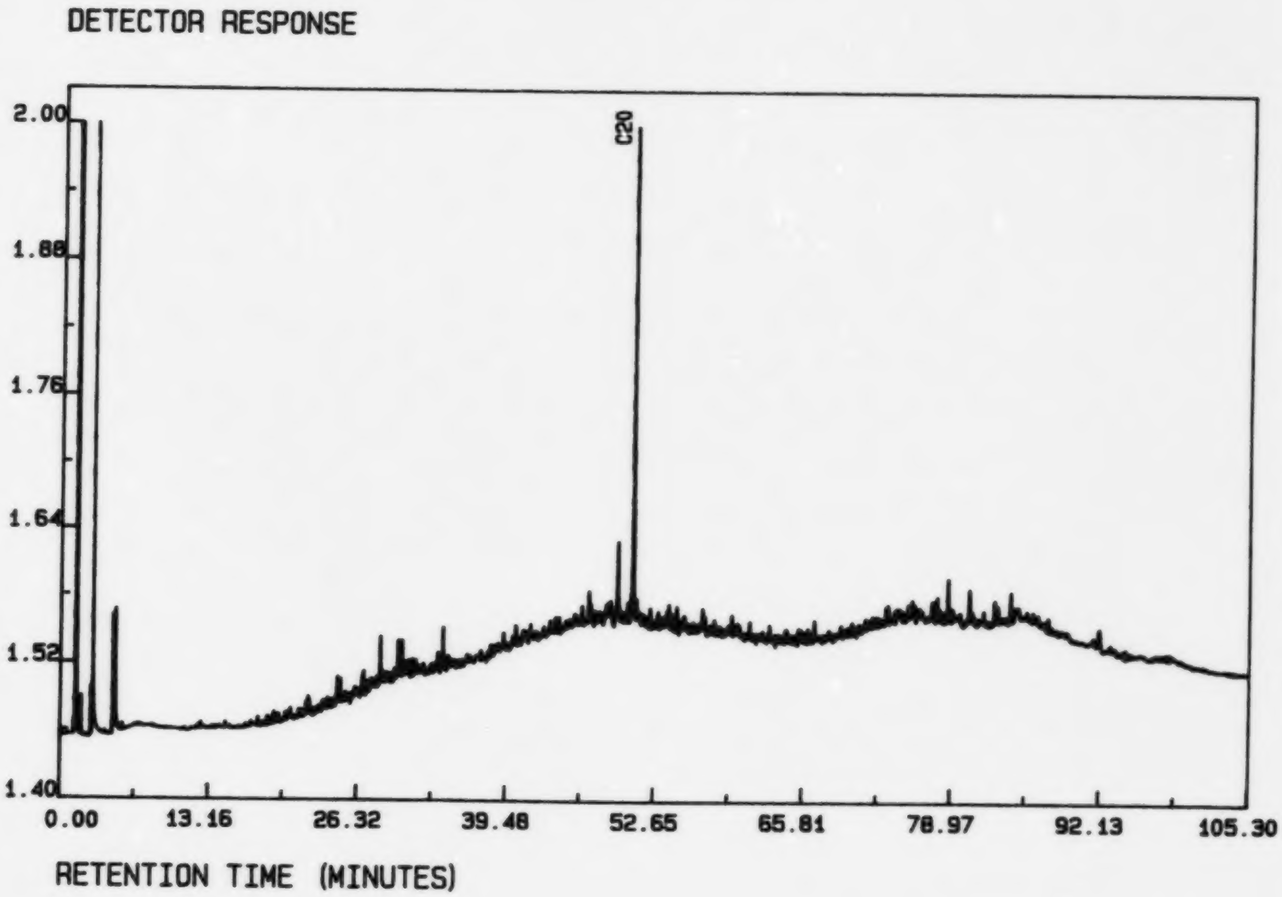


FIGURE 35

GC-FID CHROMATOGRAM OF #4 LOWER BIOREACTOR

JULY 19, 1990

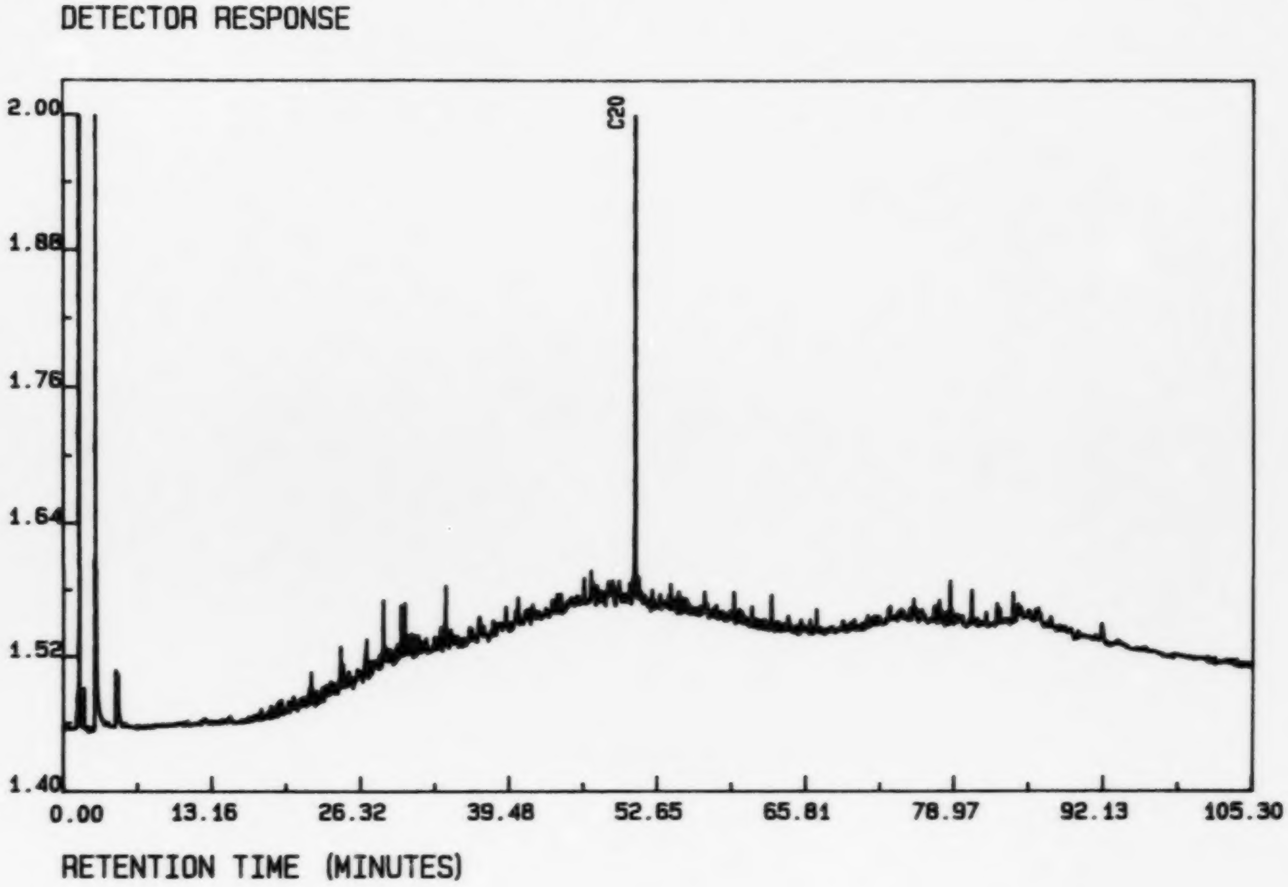


FIGURE 36

GC-FID CHROMATOGRAM OF #5 LOWER BIOREACTOR

JULY 19, 1990

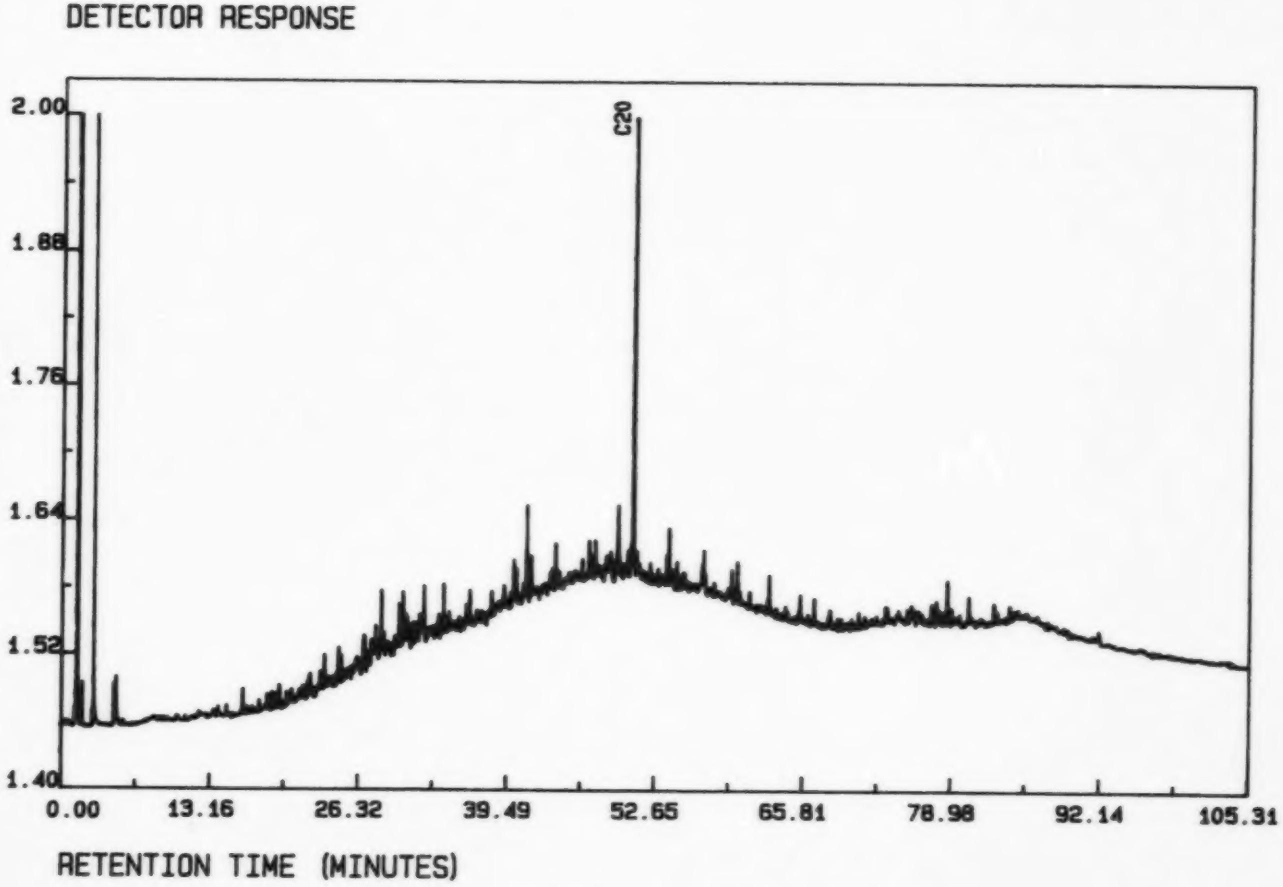


FIGURE 37
GC-FID CHROMATOGRAM OF #1 UPPER BIOREACTOR

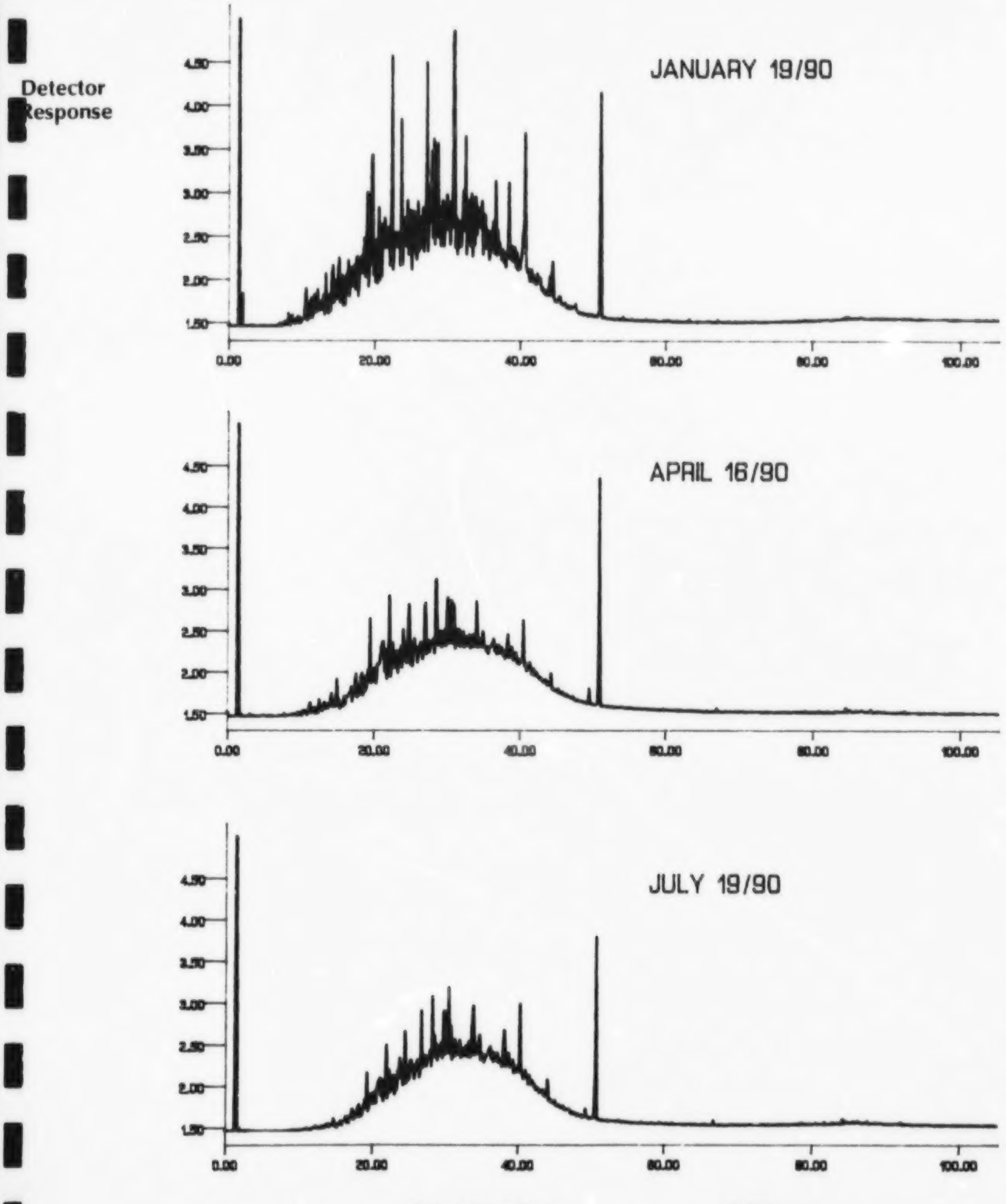


FIGURE 38
GC-FID CHROMATOGRAM OF #1 LOWER BIOREACTOR

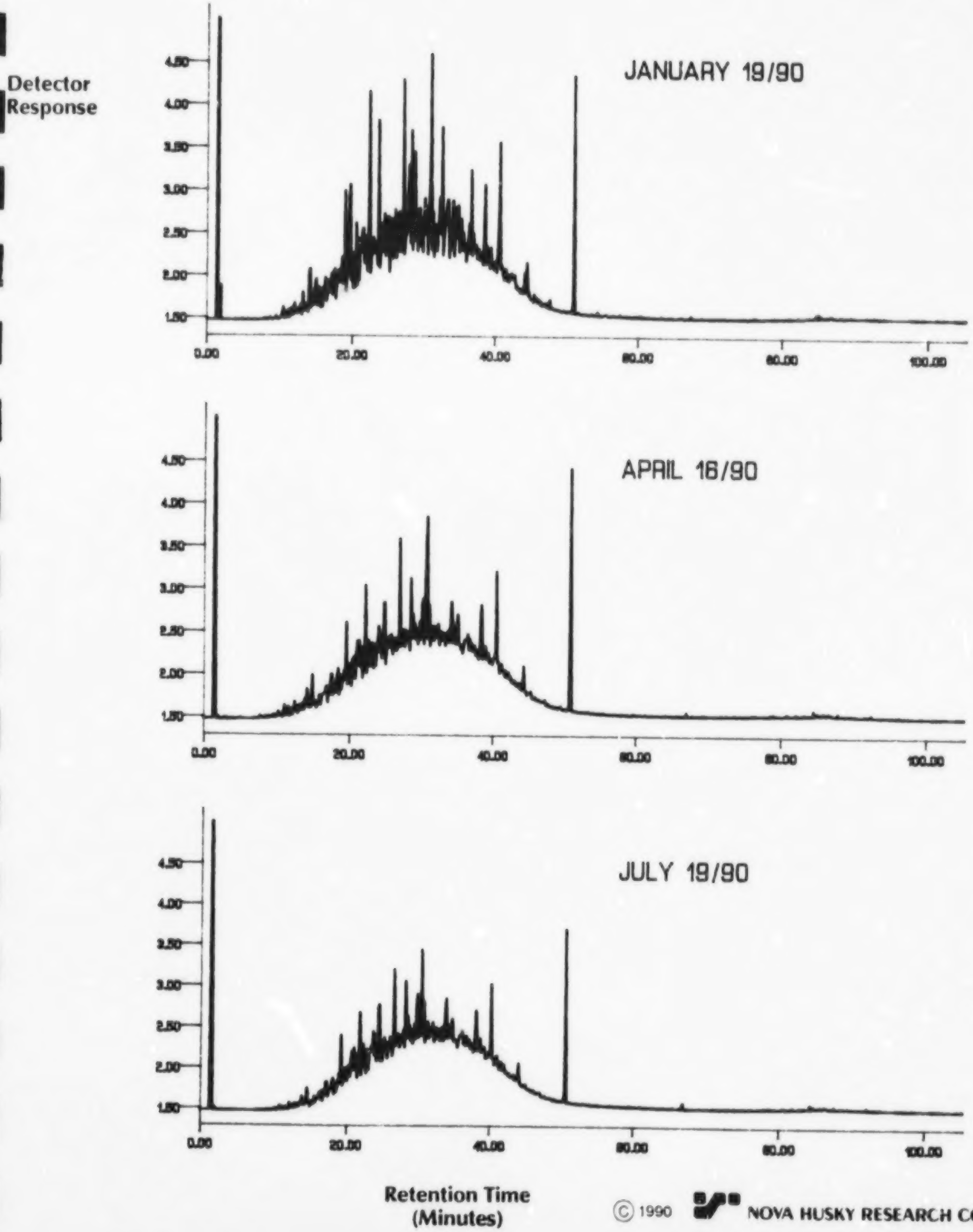
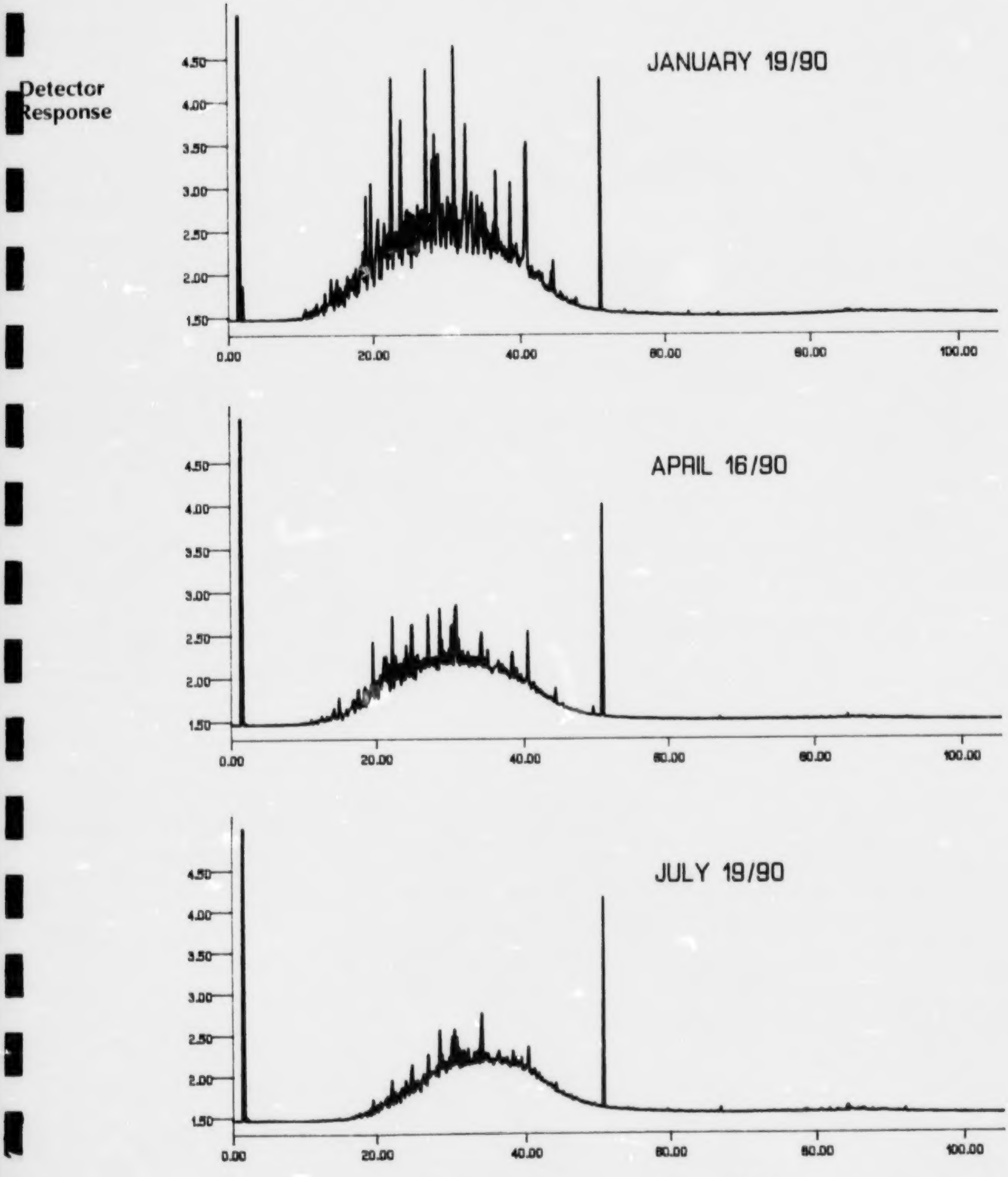


FIGURE 39

GC-FID CHROMATOGRAM OF #6 UPPER BIOREACTOR



Retention Time
(Minutes)

FIGURE 40
GC-FID CHROMATOGRAM OF #6 LOWER BIOREACTOR

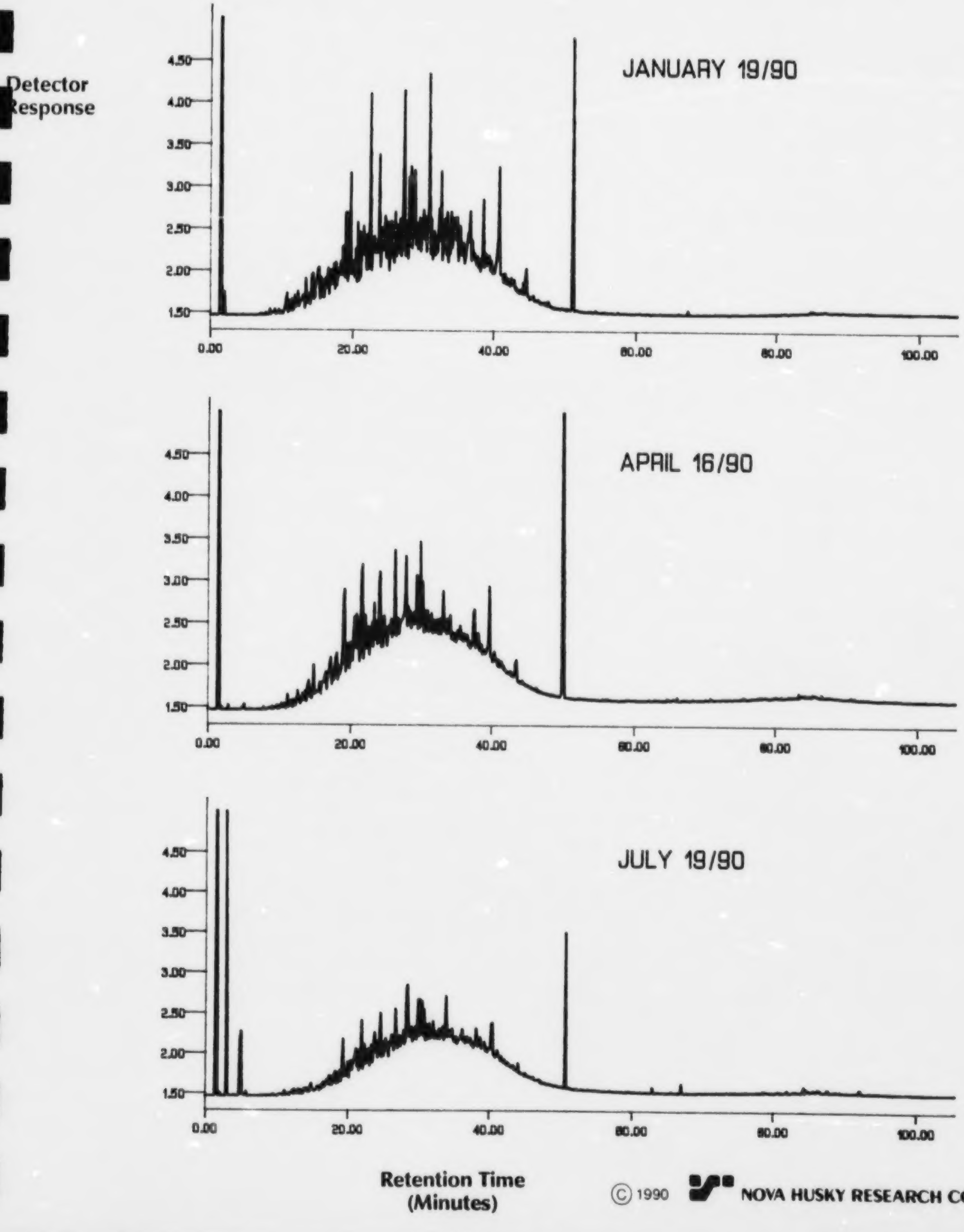


FIGURE 41

GC-FID CHROMATOGRAM OF #2 UPPER BIOREACTOR

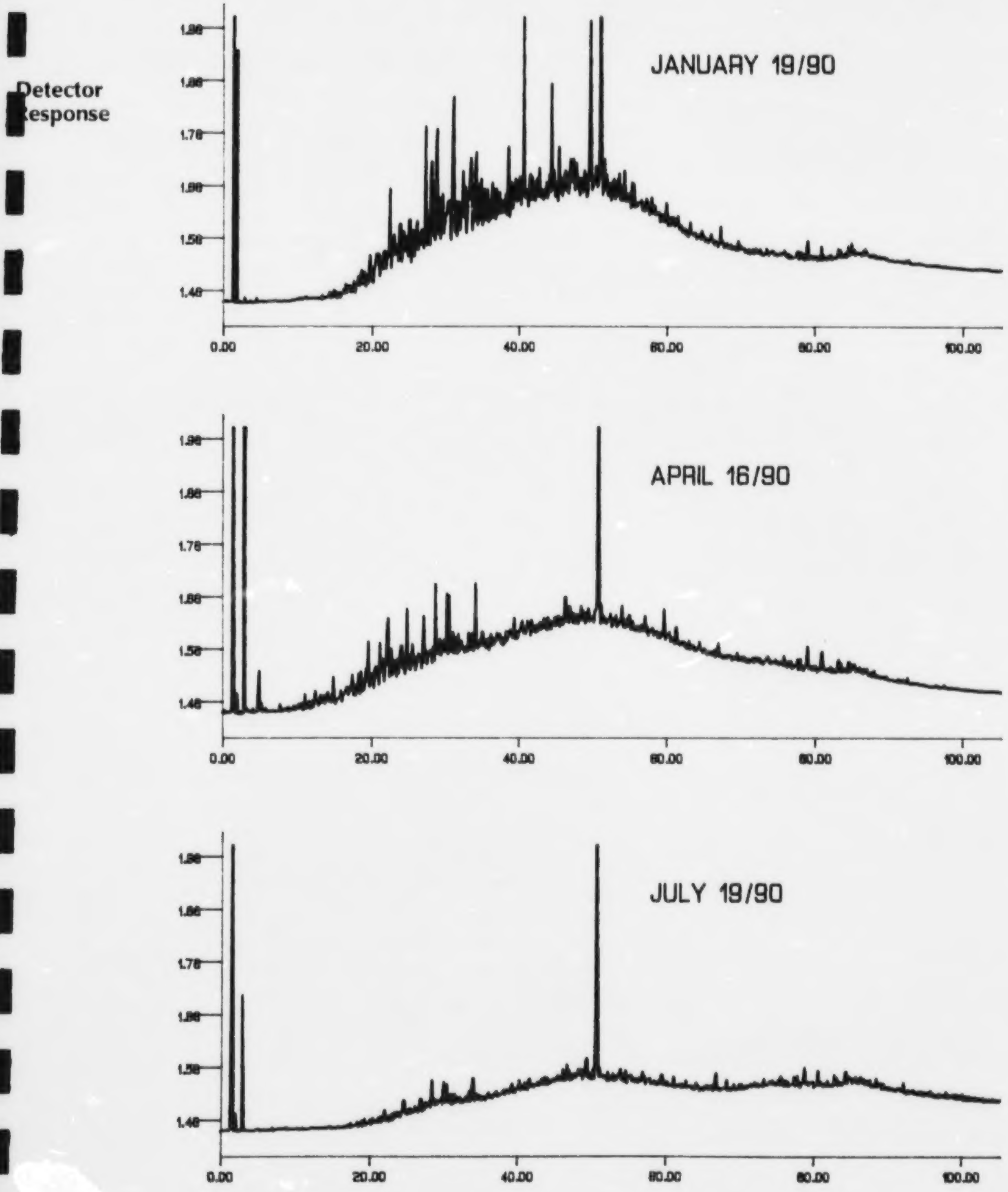


FIGURE 42

GC-FID CHROMATOGRAM OF #2 LOWER BIOREACTOR

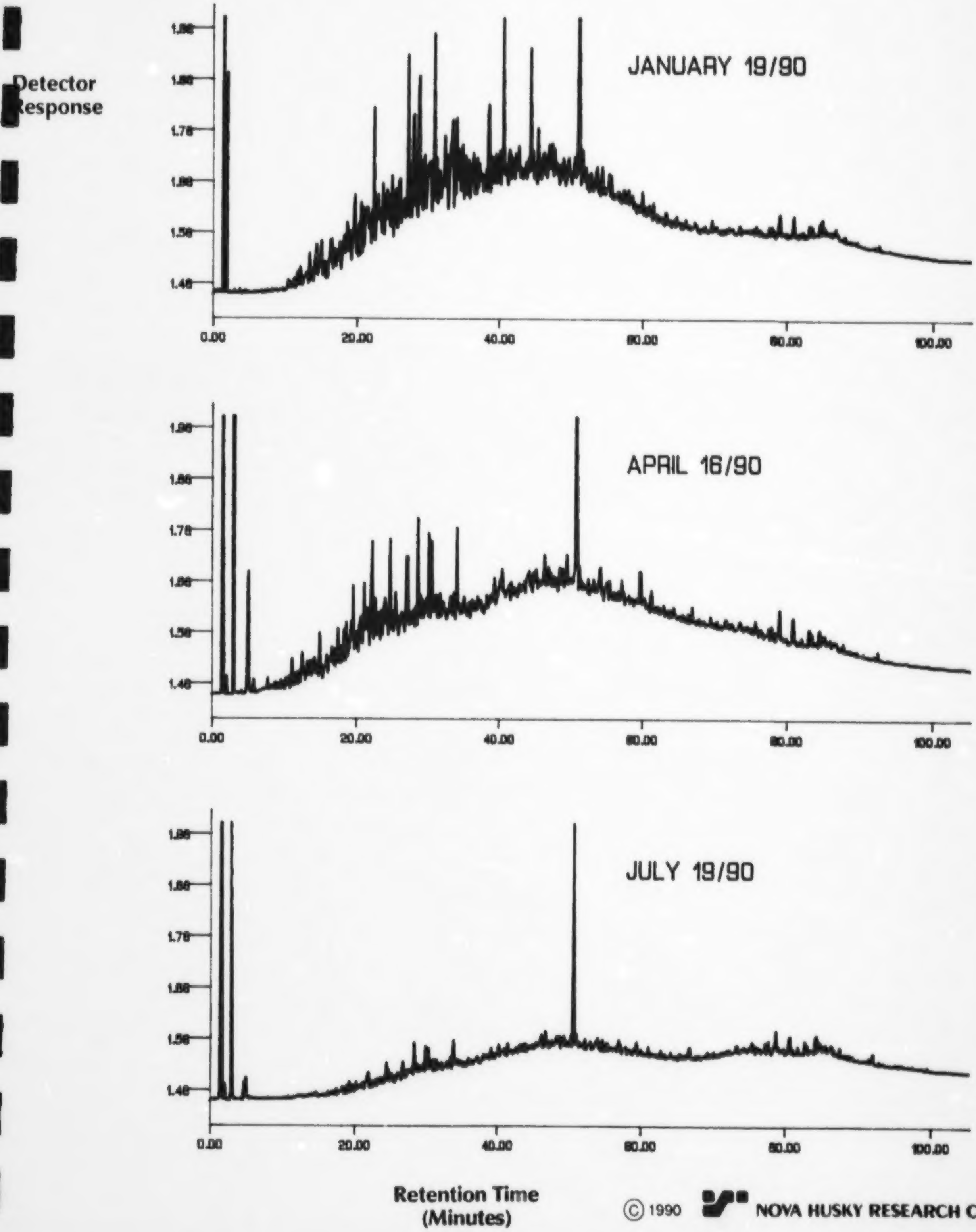


FIGURE 43

GC-FID CHROMATOGRAM OF #3 UPPER BIOREACTOR

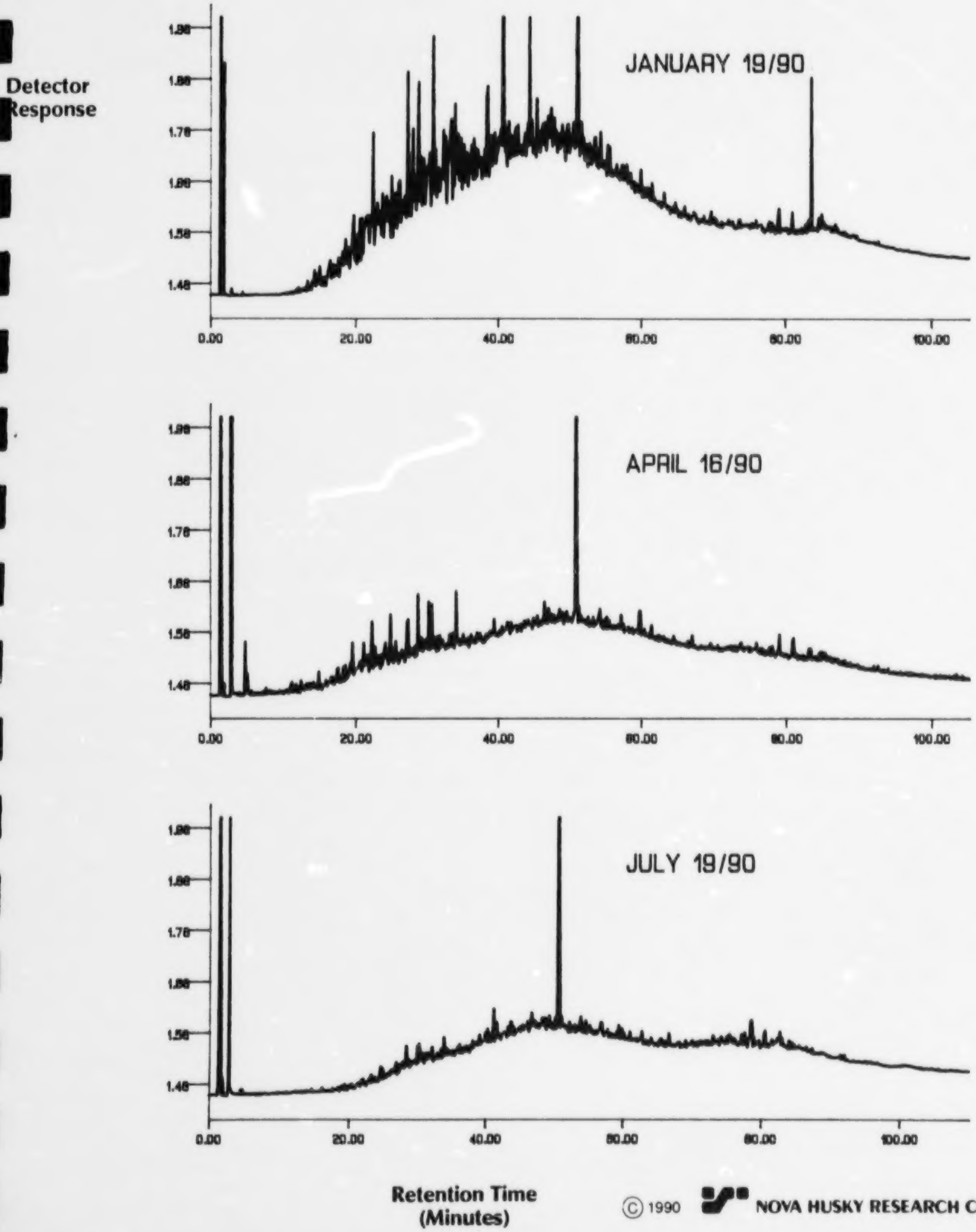


FIGURE 44

GC-FID CHROMATOGRAM OF #3 LOWER BIOREACTOR

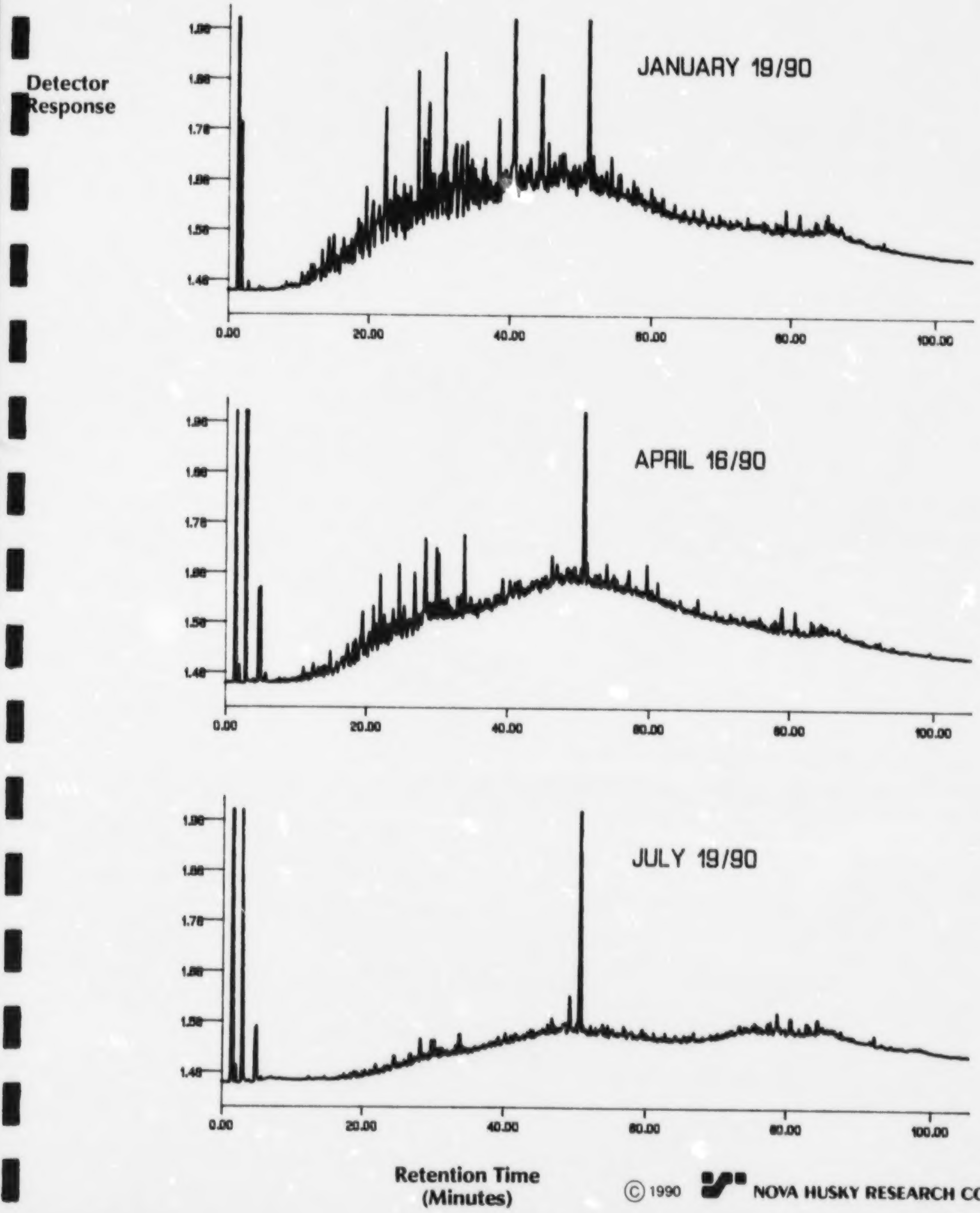


FIGURE 45
GC-FID CHROMATOGRAM OF #4 UPPER BIOREACTOR

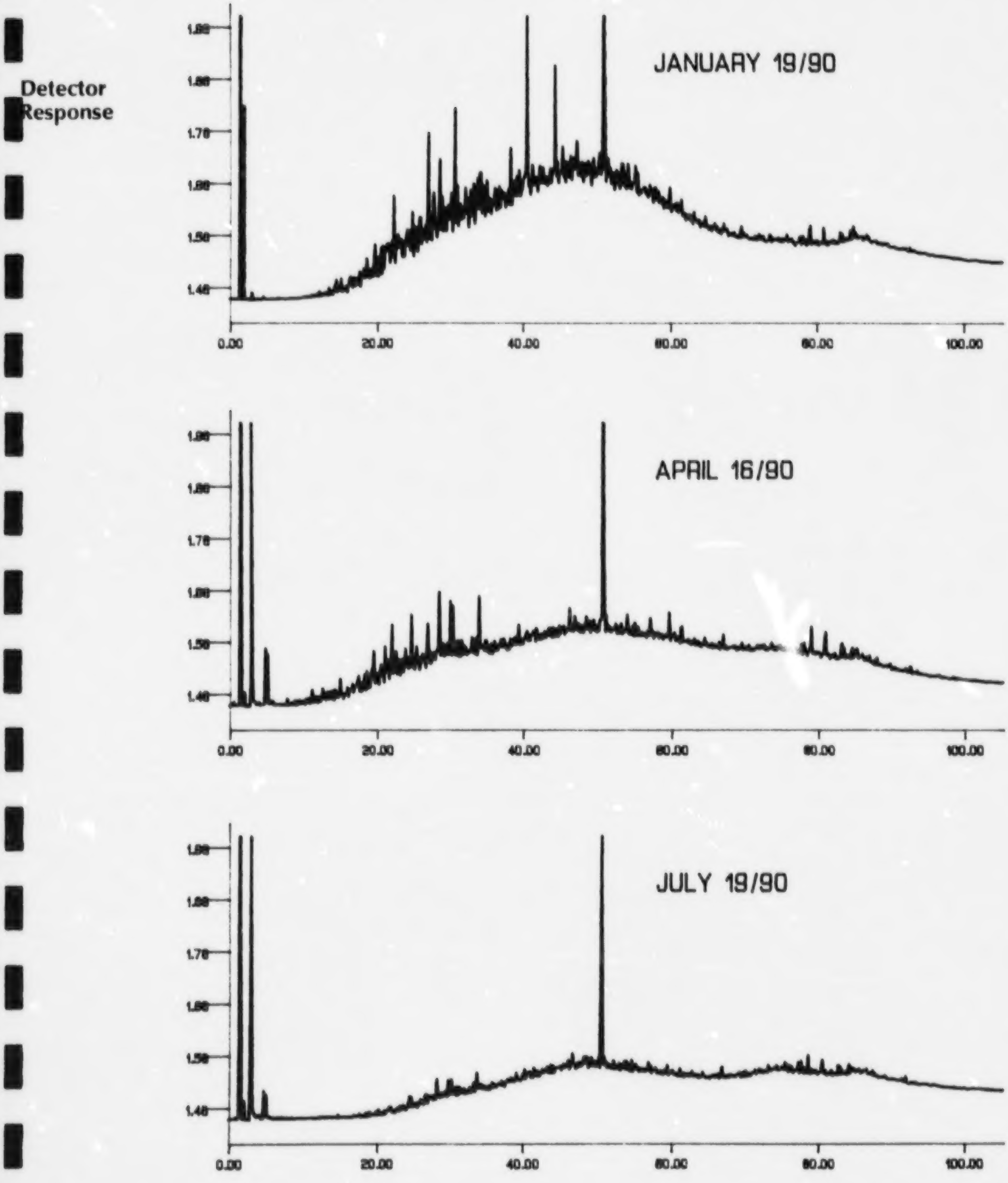


FIGURE 46

GC-FID CHROMATOGRAM OF #4 LOWER BIOREACTOR

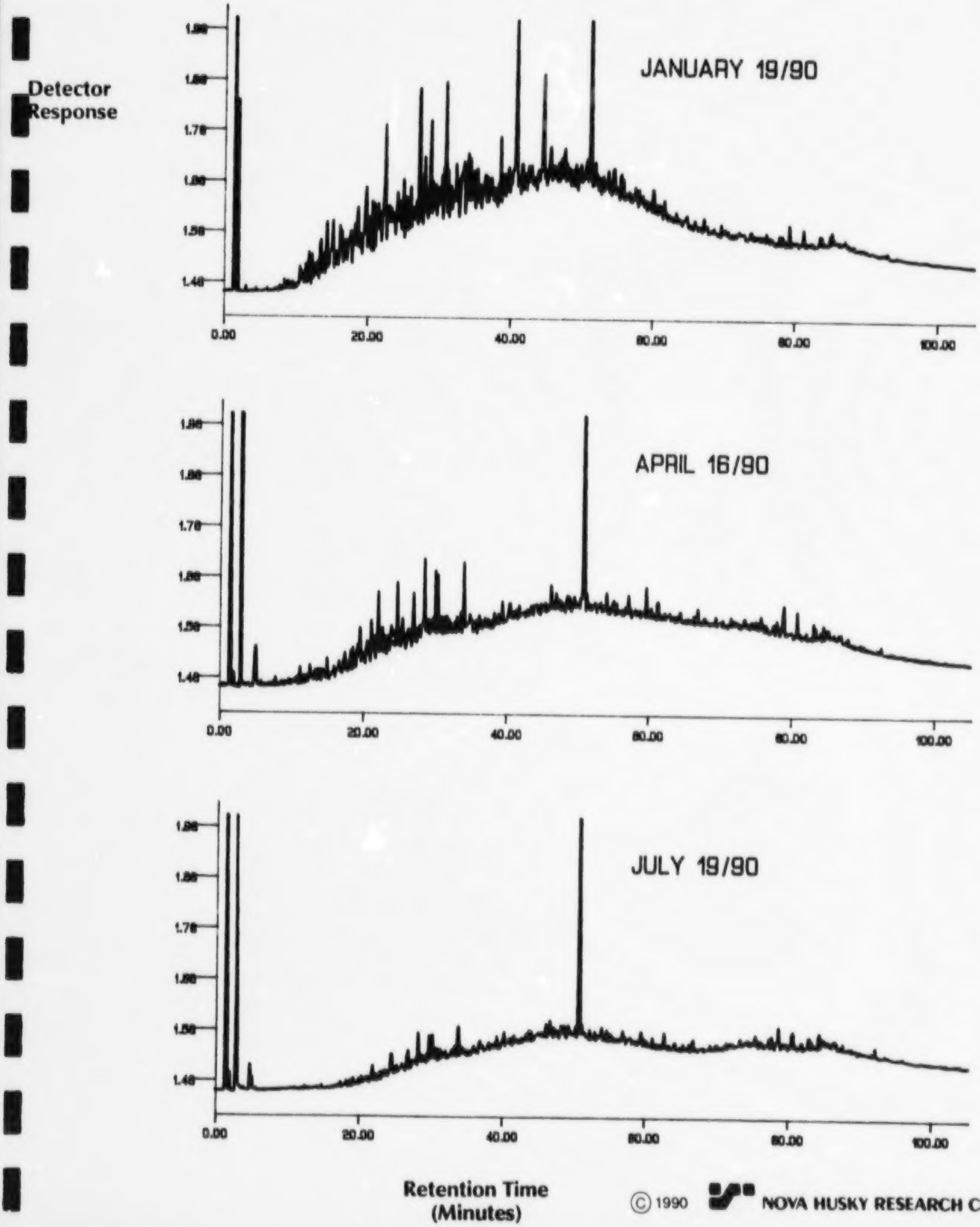
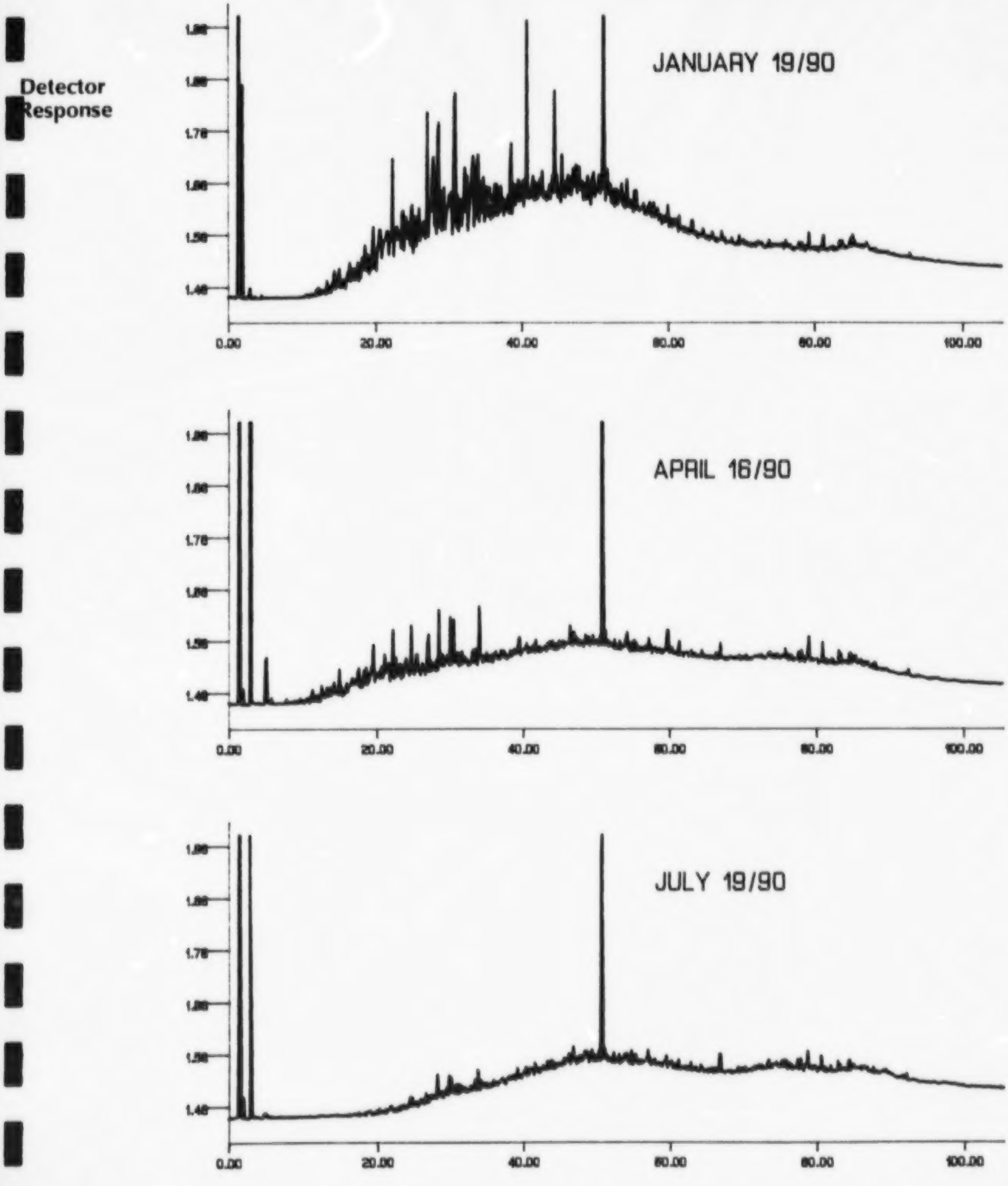


FIGURE 47

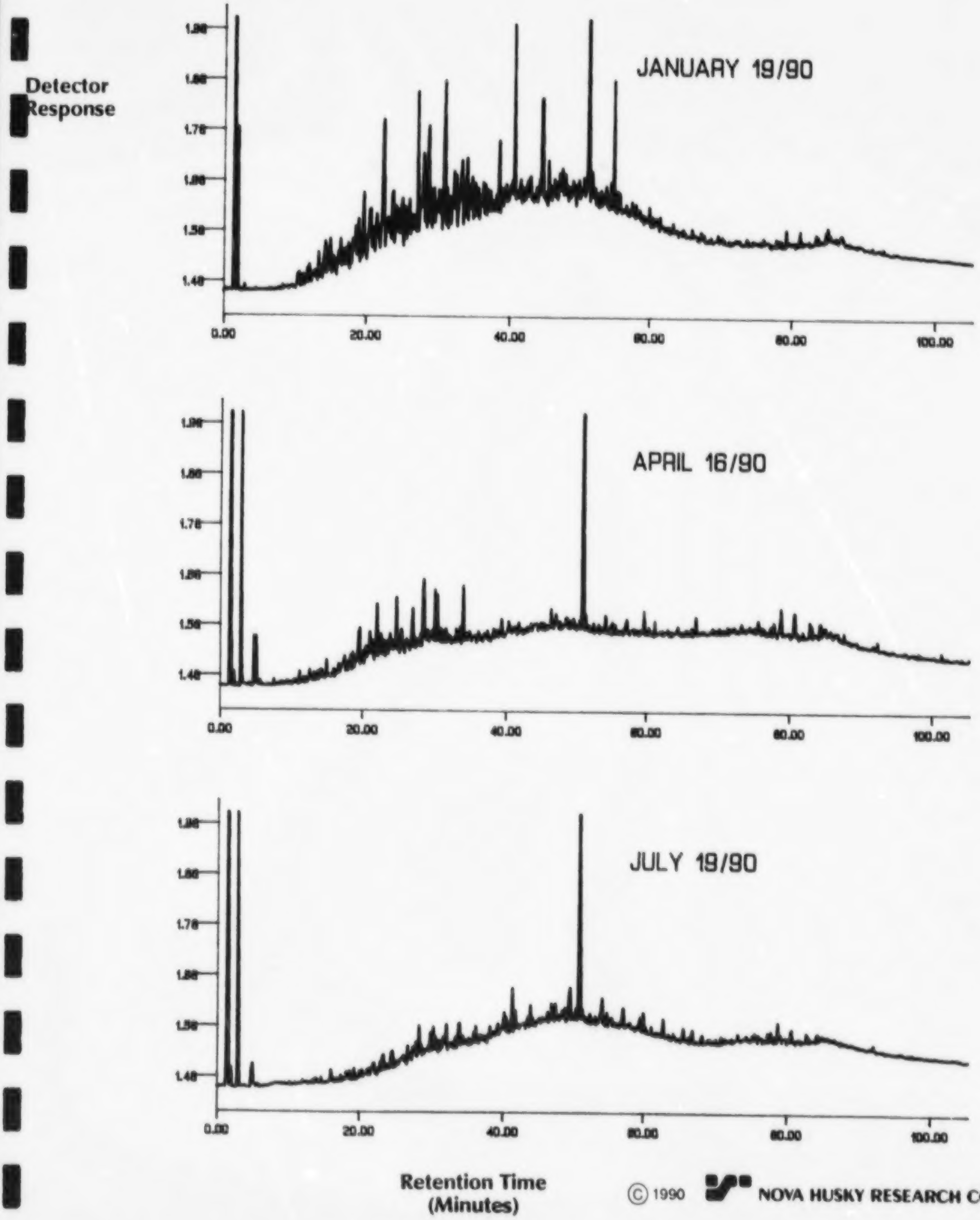
GC-FID CHROMATOGRAM OF #5 UPPER BIOREACTOR



Retention Time (Minutes)

FIGURE 48

GC-FID CHROMATOGRAM OF #5 LOWER BIOREACTOR



APPENDIX 5. LANDFILL RULES FOR ALBERTA AND SASKATCHEWAN

PROVINCE OF ALBERTA

HAZARDOUS CHEMICALS ACT

HAZARDOUS WASTE REGULATION

Alberta Regulation 505/87

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HAZARDOUS CHEMICALS ACT

(O.C. 824/87)

Approved and Ordered,
W. HELEN HUNLEY,
Lieutenant Governor.

Edmonton, December 17, 1987

Upon the recommendation of the Honourable the Minister of the Environment, the Lieutenant Governor in Council, pursuant to section 16 of the Hazardous Chemicals Act, makes the regulation in the attached Appendix, being the Hazardous Waste Regulation.

DON R. GETTY (Chairman)

APPENDIX

Hazardous Chemicals Act

HAZARDOUS WASTE REGULATION

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Schedule

Definitions

1 In this Regulation,

(a) "Act" means the *Hazardous Chemicals Act;*

(b) "carrier" means a person accepting for transportation or transporting hazardous wastes for storage, treatment or disposal;

(c) "container" means any portable device less than 454 litres in size in which a hazardous waste is stored;

(d) "empty container" means a container that contains less than 2.5 centimetres of residue remaining at the bottom of the container or less than 3% of the original contents, whichever is the lesser amount;

(e) "generator" means a person consigning hazardous wastes for storage, transportation, treatment, or disposal;

(f) "storage" means the holding of a hazardous waste for a temporary period at the end of which the hazardous waste is

(i) transported;

(ii) treated;

(iii) disposed of;

(g) "receiver" means a person receiving hazardous wastes for storage, treatment or disposal;

(h) "unrinsed empty container" means an empty container

(i) that has not been rinsed 3 times using for each rinse a clean solvent that is in an amount equal to 10% of the container volume and that is capable of removing the contained hazardous waste, or

(ii) that, in the opinion of the Director of Standards and Approvals of the Department, has not been rinsed by a method that produces results equal to or better than those produced by the method set out in subclause (i).

xemption

The following hazardous chemicals are exempt from the Act and his Regulation if they are disposed of as waste:

(a) those wastes that are not listed in Table 1 of the Schedule or defined by the criteria specified in Table 1;

(b) household wastes;

(c) agricultural wastes generated by

(i) the growing and harvesting of agricultural crops, or

(ii) the raising of animals, including animal manures returned to the soil as fertilizers;

(d) mining overburden returned to a mine site as approved pursuant to the *Land Surface Conservation and Reclamation Act* and the regulations under that Act;

(e) fly ash waste, bottom ash waste, slag waste or flue gas emission control waste generated from the combustion of domestic waste, coal, or other fossil fuels as approved pursuant to the *Clean Air Act* or the *Clean Water Act* and the regulations under those Acts;

(f) drilling fluids, produced waters, oily waste, fracture fluids, reformates, completion fluids, process and run-off waters, spent iron sponge or similar sweeteners and waste treater hay resulting from the exploration, development or production of crude oil or natural gas approved pursuant to the *Oil and Gas Conservation Act* and the regulations under that Act, the *Oil Sands Conservation Act* and regulations under that Act or the *Pipeline Act* and regulations under that Act;

(g) irrigation return flows approved pursuant to the *Water Resources Act* and the regulations under that Act;

(h) domestic sewage;

(i) any materials that are for recycling, re-use or reprocessing in facilities approved pursuant to the *Clean Water Act* or the *Clean Air Act* and the regulations under those Acts;

(j) point source discharges licensed pursuant to the *Clean Water Act* or the *Clean Air Act* and the regulations under those Acts;

(k) agricultural chemicals and their empty containers approved pursuant to the *Agricultural Chemicals Act* and the regulations under that Act;

(l) any hazardous waste, except for those containing chemicals listed in Table 2 of the Schedule, produced by a generator in an amount less than 5 kilograms if a solid, or 5 litres if a liquid, per month.

Exemption

3 Section 7.4(1)(a), (b), (d) and (e) of the Act do not apply to a person who treats, stores or disposes of hazardous wastes generated by him on premises occupied by him.

Exemption

4 Sections 7.2, 7.3 and 7.4 of the Act do not apply to a hazardous waste which is transported not more than 1 km on a highway, as defined in the *Highway Traffic Act*, by or for a generator of the hazardous waste between the properties owned or leased by the generator if

(a) the local authority, as defined in the *Transportation of Dangerous Goods Control Act*, is informed of the movement and the nature of the hazardous waste, and

(b) the person in charge of the vehicle transporting the hazardous waste displays on the vehicle a placard that corresponds to the placard set out as Figure 19 in Part II of Schedule V of the *Transportation of Dangerous Goods Regulations* (SOR 85-77) under the *Transportation of Dangerous Goods Act* (Canada).

Manifest form

5 The manifest required by the Act shall be in the form set out in the *Transportation of Dangerous Goods Regulations* (SOR 85-77) under the *Transportation of Dangerous Goods Act* (Canada).

Manifest completion

6 The applicable parts and copies of the manifest must be fully completed by the generator, carrier and receiver of hazardous wastes.

Generator's manifest duties

7 A generator shall

(a) on consigning a hazardous waste,

(i) certify the consignment on all copies of the manifest, and

(ii) have the carrier certify receipt of the waste,

(b) mail the 1st copy of the manifest to the Director of Pollution Control of the Department within 2 days, excluding Saturdays and holidays, after consigning the hazardous waste,

(c) retain the 2nd copy for at least 2 years following the consignment, and

(d) deliver the 3rd, 4th, 5th and 6th copies to the carrier.

Copies accompanying waste

8 The 3rd, 4th, 5th and 6th copies of the manifest must accompany the hazardous waste during transportation.

Carrier's manifest duties

9 The carrier shall

(a) on accepting a hazardous waste for transportation, certify the acceptance on all copies of the manifest,

(b) return the 1st and 2nd copies to the generator, and

(c) deliver the 3rd, 4th, 5th and 6th copies to the receiver.

Receiver's manifest duties

10 The receiver shall

(a) on accepting a hazardous waste for storage, treatment or disposal,

(i) certify the receipt on the 3rd, 4th, 5th and 6th copies of the manifest, and

(ii) return the 4th copy to the carrier,

(b) mail the 3rd copy to the Director of Pollution Control of the Department within 2 days, excluding Saturdays and holidays, after receiving the hazardous waste,

(c) retain the 5th copy for at least 2 years after receiving the hazardous waste, and

(d) mail the 6th copy to the generator within 2 days, excluding Saturdays and holidays, after receiving the hazardous waste.

Retained copy

11 The generator shall retain the 6th copy for at least 2 years following its receipt from the receiver.

Multiple carriers' manifest

12(1) If multiple carriers are used for 1 consignment of a hazardous waste,

(a) the generator shall fill out 1 manifest form with respect to each carrier,

(b) each carrier, on acceptance of the waste for transport, shall

(i) certify the receipt of the waste on 1 form and return copies 1 and 2 of that form to the generator, and

(ii) deliver the remaining manifest copies or remaining manifest forms or both to the receiver or next carrier,

and

(c) the receiver shall, on receiving the hazardous waste,

(i) certify the receipt of the waste on the 3rd, 4th, 5th and 6th copies of all manifest forms,

(ii) cross-reference all of the manifest,

(iii) mail the 3rd copy of each manifest form to the Director of Pollution Control of the Department within 2 days, excluding Saturdays and holidays, after receiving the waste,

(iv) mail the 4th copy of the appropriate manifest to each carrier,

(v) retain the 5th copy of each manifest for at least 2 years after receiving the waste, and

(vi) mail the 6th copy to the generator within 2 days, excluding Saturdays and holidays, after receiving the waste.

(2) A generator shall retain a copy for at least 2 years following its receipt from a receiver under subsection (1)(c).

Storage facility

13 A person who stores a hazardous waste shall, in a manner satisfactory to the Director of Standards and Approvals of the Department, store it so that

(a) any leakage is contained and prevented from entering into the remainder of the storage site and places beyond, including sewers and the ground underneath the site,

(b) the hazardous waste is adequately labelled, stating the identity of the contained waste, and

(c) the place where the hazardous waste is stored

(i) is secured from public entry,

(ii) is prominently identified as a hazardous waste storage site,

(iii) is equipped with suitable equipment to handle emergency situations, and

(iv) is provided with operators trained to respond to emergency situations specific to the substances stored.

Storage

14(1) No person shall store a hazardous waste that he did not produce unless he is a holder of a licence to operate issued under the *Clean Water Act* by the Director of Standards and Approvals of the Department.

(2) No person shall store a hazardous waste

(a) for a period exceeding 365 days, or

(b) in an amount exceeding 10 tonnes.

(3) Notwithstanding subsection (2), a hazardous waste may be stored for a period exceeding 365 days or in an amount exceeding 10 tonnes if the person who stores the hazardous waste is a holder of a licence to operate issued under the *Clean Water Act* by the Director of Standards and Approvals of the Department.

Bond

15 A person who operates a hazardous waste management facility shall provide to the Minister, if the Minister so requires, a bond in a form and in an amount that the Minister considers appropriate, and with an obligor and obligee acceptable to the Minister, to secure the performance of that person's obligations under the Act, the regulations, any agreement entered into with the Corporation and any conditions imposed by the Corporation respecting the storage, collection, transportation and treatment of hazardous wastes and the disposal of hazardous wastes and waste matter resulting from the treatment of hazardous wastes.

Unauthorized release report

16 Where an unlawful or accidental discharge, emission, escape or spill of a hazardous waste occurs at a facility for the storage, treatment or disposal of hazardous wastes, the person responsible for the hazardous waste shall, immediately on becoming aware of the occurrence, notify the Director of Pollution Control of the Department.

Landfill disposal

17(1) Hazardous wastes, except for those listed in subsection (3), may be disposed of in landfills that are approved for that purpose by the Director of Standards and Approvals of the Department.

(2) The Director of Standards and Approvals of the Department may approve the disposal of liquid hazardous wastes in a landfill that is provided with

(a) 2 liners of which at least 1 is a synthetic liner,

(b) a leachate collection and removal system,

(c) a leak detection system between the 2 liners, and

(d) a ground water monitoring system.

(3) Hazardous wastes that may not be disposed of into a landfill are

(a) liquid and solid hazardous wastes containing tri-, tetra-, and pentachlorophenol in a concentration greater than 1000 milligrams per kilogram;

(b) liquid or solid hazardous wastes containing any 1 or more of the following organic solvents in a concentration greater than 1000 milligrams per kilogram:

(i) acetone;

(ii) benzene;

(iii) n-butyl alcohol;

(iv) carbon disulfide;

(v) carbon tetrachloride;

(vi) chlorobenzene;

(vii) cresols and cresylic acid;

(viii) cyclohexanone;

(ix) ethyl acetate;

(x) ethyl benzene;

(xi) ethyl ether;

(xii) isobutanol;

(xiii) methanol;

(xiv) methylene chloride;

(xv) methyl ethyl ketone;

(xvi) methyl isobutyl ketone;

(xvii) nitrobenzene;

(xviii) 2-nitropropane;

(xix) pyridine;

(xx) tetrachloroethylene;

(xxi) toluene;

(xxii) 1,1,1 and 1,1,2-trichloroethane;

(xxiii) trichloroethylene;

(xxiv) xylene;

(c) liquid hazardous wastes containing free cyanides in excess of 1000 milligrams per kilogram;

(d) liquid hazardous wastes or earthen materials including, gravel, sand, clay and soil, that contain more than 50 parts per million by weight of polychlorinated biphenyls;

(e) hazardous wastes with a pH less than 2.0;

(f) liquid hazardous wastes containing the following contaminants in an amount equal to or greater than that shown:

(i) arsenic	500 milligrams per kilogram;
(ii) cadmium	100 milligrams per kilogram;
(iii) chromium (Cr^{+6})	500 milligrams per kilogram;
(iv) lead	500 milligrams per kilogram;
(v) mercury	100 milligrams per kilogram;
(vi) nickel	134 milligrams per kilogram;
(vii) selenium	100 milligrams per kilogram;
(viii) thallium	130 milligrams per kilogram.

Repeal

18 *The Hazardous Waste Regulation (Alta. Reg. 49/85) is repealed.*

Coming into force

19 *This Regulation comes into force on April 1, 1988.*

SCHEDULE

TABLE 1

1 Hazardous wastes listed in this Table consist of

(a) the goods, substances, and waste streams listed in List II of Schedule II in the English version of the *Transportation of Dangerous Goods Regulations* (SOR 85-77) under the *Transportation of Dangerous Goods Act* (Canada), except for those goods or substances which have the numeral "37" appearing in Column IV of that list;

(b) any container, other than an empty container, that is larger than 5 litres in capacity, and that was used to hold any goods, substances or waste streams referred to in clause (a), except for those products or substances listed in Table 2;

(c) any unrinsed empty container larger than 5 litres in capacity that was used to hold any product or substance listed in Table 2.

2 Hazardous wastes defined by criteria in this Table are the following:

(a) any mixture or solution described by Classes 2 to 9 of section 3 in the English version of the *Transportation of Dangerous Goods Regulation* (SOR 85-77) made under the *Transportation of Dangerous Goods Act* (Canada), or

(b) any mixture or solution which contains 0.01 percent by mass or greater of any of the substances listed in section 1(a) with the numerals "9.2" appearing in Column III of the list referred to in section 1(a).

TABLE 2

CHEMICALS THAT MAKE UNRINSED EMPTY CONTAINERS HAZARDOUS WASTE

Substance

Acetaldehyde, chloro-
Acetamide, N-(aminothioxomethyl)-
Acetamide 2 fluoro-
Acetic acid, fluoro-, sodium salt
Acetimidic acid, N-[(methylcarbamoyl)oxy]thio-, methyl ester
3-(alpha-Acetonylbenzyl)-4-hydroxycoumarin and salts, when present at concentrations greater than 0.3%
1-Acetyl 2-thiourea
Acrolein
Aldicarb
Aldrin
Allyl alcohol
Aluminum phosphide
5-(Aminomethyl)-3-isoxazolol
4-aAminopyridine
Ammonium picrate
Ammonium vanadate
Arsenic acid
Arsenic (III) oxide
Arsenic (V) oxide
Arsenic pentoxide
Arsenic trioxide
Arsine, diethyl-
Aziridine
Barium cyanide
Benzenamine, 4-chloro-
Benzenamine, 4-nitro-
Benzene, (Chloromethyl)
1,2 Benzenediol, 4-[1-hydroxy-2 (methylamino)ethyl]
Benzenethiol
Benzyl chloride
Beryllium dust
Bis(chloromethyl) ether
Bromoacetone
Brucine
Calcium cyanide
Camphene, octachloro-
Carbamimidoselenoic acid
Carbon bisulfide
Carbon disulfide
Carbonyl chloride
Chlorine cyanide
Chloroacetaldehyde
p-Chloroaniline
1-(o-Chlorophenyl)thiourea
3-Chloropropionitrile
Copper cyanides
Cyanides (soluble cyanide salts), not elsewhere specified
Cyanogen
Cyanogen chloride
Dichlorophenylarsine
Dieldrin
Diethylarsine
O,O-Diethyl S [2 (ethylthio)ethyl] phosphorodithioate

Substance

Diethyl-p-nitrophenyl phosphate
O,O Diethyl O-pyrazinyl phosphorothioate
Diisopropyl fluorophosphate
Dimethoate
3,3-Dimethyl-1-(methylthio)-2-butanone, O-[(methylamine)carbonyl] oxime
O,O-Dimethyl O-p nitrophenyl phosphorothioate
Dimethylnitrosamine
alpha, alpha-Dimethylphenethylamine
4,6-Dinitro-o-cresol and salts
4,6-Dinitro-o-cyclohexylphenol
2,4-Dinitrophenol
Dinoseb
Diphosphoramide, octamethyl-
Disulfoton
2,4-Dithiobiuret
Dithiopyrophosphoric acid, tetraethyl ester
Endosulfan
Endothall
Endrin
Epinephrine
Ethanamine, 1,1-dimethyl-2-phenyl
Ethenamine, N-methyl-N-nitroso-
Ethyl cyanide
Ethylenimine
Famphur
Fluorine
Fluoroacetamide
Fluoroacetic acid, sodium salt
Fulminic acid, mercury (II) salt
Heptachlor
1,2,3,4,10,10-Hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-endo, endo-1,4:5,8-dimethanonaphthalene
1,2,3,4,10,10-Hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-endo, exo-1,4:5,8-demethanonaphthalene
1,2,3,4,10,10-Hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-endo, endo-dimethanonaphthalene
1,2,3,4,10,10-Hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-endo, exo-dimethanonaphthalene
Hexachlorohexahydro-exo, exo-dimethanonaphthalene
Hexaethyl tetraphosphate
Hydrazinecarbothioamide
Hydrazine, methyl-
Hydrocyanic acid
Hydrogen cyanide
Hydrogen phosphide
Isocyanic acid, methyl ester
3(2H)-Isoxazolone, 5-(aminomethyl)
Mercury, (acetato-O)phenyl-
Mercury fulminate
Methane, oxybis(chloro-

TABLE 2 (cont'd)

CHEMICALS THAT MAKE UNRINSED EMPTY CONTAINERS HAZARDOUS WASTE

Substance

Methane, tetranitro-
Methanethiol, trichloro-
4,7-Methano-1H-indene, 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-
Methomyl
2-Methylaziridine
Methyl hydrazine
Methyl isocyanate
2-Methyllactonitrile
Methyl parathion
alpha-Naphthylthiourea
Nickel carbonyl
Nickel cyanide
Nickel (II) cyanide
Nickel tetracarbonyl
Nicotine and salts
Nitric Oxide
p-Nitroaniline
Nitrogen dioxide
Nitrogen (II) oxide
Nitrogen (IV) oxide
Nitroglycerine
N-Nitrosodimethylamine
N-Nitrosomethylvinylamine
5-Norbornene-2,3-dimethanol, 1,4,5,6,7,7-hexachloro, cyclic sulfite
Octamethylpyrophosphoramide
Osmium oxide
Osmium tetroxide
7-Oxabicyclo [2.2.1] heptane-2,3-dicarboxylic acid
Parathion
Phenol, 2-cyclohexyl-4,6-dinitro
Phenol, 2,4-dinitro-
Phenol, 2,4-dinitro-6-methyl-
Phenol, 2,4 dinitro-6-(1-methylpropyl)-
Phenol, 2,4,6-trinitro, ammonium salt
Phenyl dichloroarsine
Phenylmercuric acetate
N-Phenylthiourea
Phorate
Phosgene
Phosphine
Phosphoric acid, diethyl p-nitrophenyl ester
Phosphorodithioic acid, O,O-dimethyl S [2 (methylamino) 2-oxoethyl] ester
Phosphorofluoric acid, bis(1 methyl ethyl)-ester
Phosphorothioic acid, O,O diethyl S (ethylthio)methyl ester
Phosphorothioic acid, O,O diethyl O (p-nitrophenyl) ester
Phosphorothioic acid, O,O-diethyl O pyrazinyl ester

Substance

Phosphorothioic acid, O,O-dimethyl-O-[p-((dimethylamino-sulfonyl) phenyl)] ester
Plumbane, tetraethyl-
Potassium cyanide
Potassium silver cyanide
Propanal, 2-methyl-2-(methylthio)-, O[(methylamino)Carbonyl]oxime
Propanenitrile
Propanenitrile, 3-chloro-
Propanenitrile, 2-hydroxy-2-methyl-
1,2,3-Propanetriol, trinitrate-
2-Propanone, 1-bromo-
Propargyl alcohol
2-Propenal
2 Propen 1-ol
1,2 Propylenimine
2-Propyn-1-ol
4-Pyridinamine
Pyridine, (S)--3-(1-methyl-2-Pyrrolidinyl)-, and salts
Pyrophosphoric acid, tetraethyl ester
Selenourea
Silver cyanide
Sodium azide
Sodium cyanide
Strontium sulfide
Strychnidin-10 one, and salts
Strychnidin-10-one, 2,3,-dimethoxy-
Strychnine and salts
Sulfuric acid, thallium(1) salt
Tetraethyldithiopyrophosphate
Tetraethyl lead
Tetraethylpyrophosphate
Tetranitromethane
Tetraphosphoric acid, hexaethyl ester
Thallic oxide
Thallium (III) oxide
Thallium (I) selenite
Thallium (I) sulfate
Thiofanox
Thioimidodicarbonic diamide
Thiophenol
Thiosemicarbazide
Thiourea (2-chlorophenyl)-
Thiourea, 1 naphthalenyl-
Thiourea, phenyl-
Toxaphene
Trichloromethanethiol
Vanadic acid, ammonium salt
Vanadium pentoxide
Vanadium (V) oxide
Warfarin, when present at concentrations greater than 0.3%
Zinc cyanide
Zinc Phosphide, when present at concentrations greater than 10%

REVISED REGULATIONS OF SASKATCHEWAN

The Regulations printed in this Part of the Gazette form a part of *The Revised Regulations of Saskatchewan* which are being compiled over the next few years. This part contains revised regulations and amendments to revised regulations. Amendments to existing regulations, which are not yet revised, are printed in Part III.

CHAPTER E-10.2 REG 4

The Environmental Management and Protection Act
Subsection 38(1)

Order in Council 701/86, dated June 25, 1986.

(Filed June 26, 1986)

ORDER

His Honour the Lieutenant Governor in Council, on the recommendation of the Minister of the Environment, pursuant to subsection 38(1) of *The Environmental Management and Protection Act*, makes *The Municipal Refuse Management Regulations* in accordance with the attached Schedule.

SCHEDULE

1 These regulations may be cited as *The Municipal Refuse Management Regulations*. Title

2 In these regulations: Interpretation

(a) "Act" means *The Environmental Management and Protection Act*; "Act"

(b) "dispose" means to place, destroy by any means or otherwise get rid of; "dispose"

(c) "garbage" means putrescible animal and vegetable remains and by-products resulting from the handling, preparation, cooking and consumption of food; "garbage"

(d) "liquid domestic sewage" means any remains or by-products which contain animal, mineral or vegetable matter in solution or suspension; "liquid domestic sewage"

(e) "modified landfill practices" means practices that comply with subsections 7(2) and (4); "modified landfill practices"

(f) "permit" means a valid and subsisting permit issued pursuant to section 7; "permit"

(g) "putrescible" means capable of being putrid; "putrescible"

(h) "refuse" means: "refuse"

(i) remains;

(ii) by-products; and

(iii) discarded materials;

resulting from domestic, commercial, industrial or agricultural activities and includes garbage, rubbish, street cleanings and yard clippings but does not include liquid domestic sewage;

"rubbish"

(i) "rubbish" means nonputrescible remains and by-products, consisting of both combustible and non-combustible remains and by-products, and includes paper, cardboard, tin cans, wood, glass, bedding and crockery;

"sanitary landfill practices"

(j) "sanitary landfill practices" means practices that comply with subsections 7(2) and (3);

"scavenging system"

(k) "scavenging system" means a system for the collection of refuse;

"transfer station"

(l) "transfer station" means a site at which refuse is concentrated or accumulated for transportation to a waste disposal ground;

"waste disposal ground"

(m) "waste disposal ground" means a site at which refuse is disposed of.

Scavenging systems and waste disposal grounds required

3(1) The council of every municipality other than a rural municipality or a northern settlement shall provide:

(a) a scavenging system for the collection and disposal of refuse within the municipality; and

(b) a waste disposal ground for the disposal of refuse mentioned in clause (a).

(2) The council of every rural municipality and the local advisory committee of every northern settlement shall provide:

(a) a scavenging system for the collection and disposal of refuse:

(i) from each hamlet or subdivision within the municipality or within the northern settlement, as the case may be; and

(ii) from each other area of the municipality or the northern settlement in which ten or more buildings are located, in the opinion of the minister, in close proximity to each other and are occupied from time to time for recreational purposes; and

(b) a waste disposal ground for the disposal of refuse mentioned in clause (a).

(3) The scavenging system and waste disposal ground mentioned in subsections (1) and (2) may be a joint scavenging

system or a joint waste disposal ground operated jointly by more than one municipality.

Compliance with regulations

4 Every municipality shall take those steps that are necessary in the circumstances to ensure that these regulations are complied with by any person with whom the municipality has entered into an agreement to provide a scavenging system or to operate and maintain a waste disposal ground.

Prohibition

5 No person shall operate or establish a waste disposal ground:

(a) without a permit; and

(b) unless he is the owner of the land on which the waste disposal ground is situated or has acquired the right to use the land for that purpose.

Permit applications

6(1) In this section, "cell" means the area of a waste disposal ground where spreading, compacting and covering of refuse is conducted.

(2) Every applicant for a permit shall apply on a form provided by the minister and shall forward with his application the information and material specified in this section.

(3) An application for a permit authorizing solid wastes to be disposed of in a waste disposal ground serving a municipality of more than 5,000 persons or a number of municipalities with an aggregate population of more than 5,000 persons is to be accompanied by one or more topographic maps of the proposed site at a topographic map scale of 1:5000 with a maximum of two metre contour intervals, and shall indicate:

(a) the location and boundaries of the proposed site including the legal description of the land;

(b) the proposed land fill area;

(c) the projected life of the landfill;

(d) any borrow area;

(e) a typical cross-section of each proposed cell;

(f) the access road;

(g) the on-site road;

(h) the grades for proper drainage;

(i) the proposed fencing;

(j) the site security, proposed hours of operation, method for control of litter on adjacent land or waterbodies;

(k) the special drainage structures, if necessary;

(l) the location and depth of any wells and the location of rivers, lakes, streams or other natural bodies of water within one kilometre of the proposed site;

(m) the surficial geology, water table, hydraulic gradients, and baseline ground water quality in the area;

(n) the location of any irrigation ditch or other irrigation works within one kilometre of the proposed site;

(o) the location of any streets, roads, highways or railway rights-of-way within 100 metres of the proposed site;

(p) any building or structure on or within 500 metres of the proposed site;

(q) the existing and proposed utilities, pipelines or easements;

(r) an estimate of daily volumes or weights of solid waste;

(s) the existing or proposed land use activities within a one kilometre radius of the proposed site; and

(t) any other information that is required by the minister.

(4) An application for a permit authorizing solid wastes to be disposed of in a waste disposal ground serving a municipality of fewer than 5,000 persons or a number of municipalities with an aggregate population of fewer than 5,000 persons is to be accompanied by a sketch plan showing:

(a) the location and boundaries of the proposed site, including the legal description of the land on which the proposed site is to be located and of the proposed fencing for the site;

(b) the general slope of the ground in the vicinity;

(c) the location and depth of any wells within a one kilometre radius of the proposed site;

(d) the location of any rivers, lakes, streams, irrigation works or other natural bodies of water within one kilometre of the proposed site;

(e) the surficial geology, water table and hydraulic gradients in the area;

(f) the location of any streets, roads, highways or railway rights-of-way within 100 metres of the proposed site;

(g) any buildings or structures on or within 500 metres of the proposed site;

(h) the existing or proposed utilities, pipelines or easements found on or traversing the site;

(i) the existing or proposed land use activities within a one kilometre radius of the proposed site; and

(j) any other information required by the minister.

Issuance

7(1) Where the minister receives an application pursuant to section 6 and is satisfied that the applicant has complied with the Act and these regulations, he may issue a permit authorizing the applicant to establish, operate or maintain a waste disposal ground and shall state in every permit that authorizes the establishment, operation or maintenance of a waste disposal ground whether the waste disposal ground is to be operated in accordance with modified landfill practices or sanitary landfill practices.

(2) Where refuse is disposed of at a waste disposal ground, it is to be disposed of by:

(a) spreading the refuse;

(b) compacting the refuse in layers;

(c) covering the layers with soil approximately one quarter of the depth of the refuse layer;

(d) covering the soil mentioned in clause (c) with .5 metres of compacted material; and

(e) using a cover material that shall be free of putrescible material and large objects.

(3) Where a permit authorizes the establishment, operation or maintenance of a waste disposal ground in accordance with sanitary landfill practices, the permit holder shall cause the procedures described in subsection (2) to be carried out not less frequently than three times per week.

(4) Where a permit authorizes the establishment, operation or maintenance of a waste disposal ground in accordance with modified landfill practices, the permit holder shall cause the procedures described in subsection (2) to be carried out not less frequently than the frequency specified in his permit.

(5) The minister may impose any terms and conditions on a permit:

(a) at the time the permit is issued; or
(b) at any time after the permit is issued;

and may amend, vary, revoke or replace those terms and conditions.

(6) No person to whom a permit is issued pursuant to this section shall fail to comply with the terms and conditions of the permit.

Location of waste disposal grounds

8(1) In this section, "highway" means a road, parkway, driveway, square or place designed and intended for or used by the general public for the passage of vehicles, but does not include any area, whether privately or publicly owned, that is primarily intended to be used for the parking of vehicles and the necessary passageways on that area.

(2) Unless the permit states otherwise, a waste disposal ground is not to be located:

(a) within 500 metres of:

(i) a restaurant, canteen, cafeteria or any other place where food is cooked or prepared for human consumption or is consumed;

(ii) motel, hotel, domestic residence or any other building occupied as a residence; or

(iii) a school, church, community centre or recreation facility or similar public building; or

(b) within 100 metres of a highway or a cemetery within the meaning of *The Cemeteries Act*.

Burning of refuse

9(1) Subject to subsection (2), no person shall dispose of refuse by burning at a waste disposal ground.

(2) Refuse may be disposed of at a waste disposal ground by burning where burning of refuse is authorized in the permit issued with respect to the waste disposal ground.

(3) Where refuse is disposed of by burning pursuant to subsection (2), the burning shall be carried out in accordance with:

(a) any conditions specified in the permit; and

(b) any other applicable Act or law.

Prohibition re disposal

10(1) Subject to subsection (2), no person shall, at a waste disposal ground, dispose of:

(a) automobiles or other large metal objects;

(b) pesticides and other hazardous wastes, or any material or thing treated with any of those substances, including the receptacles containing them;

(c) petroleum, petroleum remains or by-products;

(d) refuse that is the remains or by-product of slaughter house operations; or

(e) liquid domestic sewage.

(2) The minister may, in writing, authorize the disposal at a waste disposal ground, on any terms or conditions he considers advisable, of materials or things mentioned in subsection (1).

(3) No person who, pursuant to subsection (2), is disposing of materials or things mentioned in subsection (1) shall fail to comply with the terms and conditions specified by the minister.

Transfer stations

11 No person shall establish, construct or operate a transfer station without the prior written approval of the minister.

Blowing of refuse

12 Every person who holds a permit to establish or operate a waste disposal ground shall take all reasonable steps in the circumstances to:

(a) prevent refuse from blowing away from the site; and

(b) retrieve refuse that has blown away or otherwise left the site.

Closure of waste disposal ground

13(1) Before a waste disposal ground is to be closed, the holder of the permit respecting the waste disposal ground shall:

(a) submit a proposal to the minister with respect to closing the waste disposal ground that outlines the steps proposed to be taken to protect the environment; and

(b) obtain the prior written approval of the minister to do so.

(2) The proposal mentioned in subsection (1) is to be in the form and contain that information that the minister may require.

(3) The minister may approve the proposal to close the waste disposal ground and impose any terms and conditions that he considers appropriate to ensure long-term protection of the environment on the approval.

(4) A person mentioned in subsection (1) shall when closing the waste disposal ground:

(a) take whatever steps are necessary in the circumstances to protect the environment; and

(b) comply with any terms or conditions specified by the minister pursuant to subsection (3).

Transportation of sewage

14(1) No person shall carry on the business of transporting and disposing of liquid domestic sewage without a permit issued by the minister authorizing him to do so.

(2) An applicant for a permit pursuant to this section shall:

(a) apply on a form provided by the minister; and

(b) supply the minister with any information that he may require.

(3) Where the minister has received an application pursuant to this section and is satisfied that the applicant has complied with this section, he may issue a permit authorising the holder to carry on the business of transporting and disposing of liquid domestic sewage.

(4) The minister may impose any terms and conditions on a permit issued pursuant to this section:

(a) at the time the permit is issued; or

(b) at any time after the permit is issued;

and may amend, vary, revoke or replace those terms and conditions.

(5) No person to whom a permit is issued pursuant to this section shall fail to comply with the terms and conditions of the permit.

Exemption

15(1) Notwithstanding any other provision of these regulations, and where the minister considers it to be not contrary to the public interest, he may, on his own initiative or on the application of any person who is in his opinion interested in the matter, by order exempt a person from all or any provision of these regulations.

(2) The minister may impose any terms and conditions on an order made pursuant to subsection (1) that he considers appropriate.

(3) No person to whom an order made pursuant to subsection (1) relates shall fail to comply with any terms or conditions imposed on the order.

Cancellation, amendment

16(1) Subject to subsection (3), where:

(a) a person to whom a permit or a permit issued pursuant to section 14 has failed to comply with the Act, these regulations or any provision or term or condition of his permit; or

(b) in the opinion of the minister, it is appropriate and in the public interest;

the minister may cancel the permit or the permit issued pursuant to section 14, as the case may be.

(2) Subject to subsection (3), where the minister considers it appropriate, he may amend a permit or a permit issued pursuant to section 14.

(3) The minister shall not cancel or amend pursuant to this section a permit or a permit issued pursuant to section 14 without giving the holder of the permit or the permit issued pursuant to section 14, as the case may be, an opportunity to be heard, unless, in the minister's opinion, it is necessary to cancel immediately in which case he:

(a) may immediately cancel or amend the permit or the permit issued pursuant to section 14; and

(b) shall give the holder of the permit or the permit issued pursuant to section 14 immediate written notice of the cancellation or amendment and give him an opportunity to be heard within 15 days of the cancellation or amendment.

Coming into force

17 These regulations come into force on the day on which they are filed with the Registrar of Regulations.